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HEADQUARTERS
U.S. ARMY ARMAMENT,
MUNITIONS AND CHEMICAL COMMAND

FINAL REPORT

DEVELOPMENT OF
METHODOLOGY AND TECHNOLOGY
FOR
IDENTIFYING AND QUANTIFYING
EMISSION PRODUCTS
FROM
OPEN BURNING AND OPEN DETONATION
THERMAL TREATMENT METHODS.

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FIELD TEST SERIES A, B, AND C

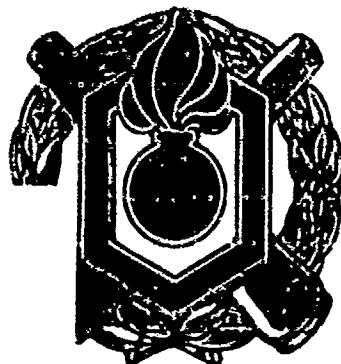
VOLUME 2, PART A
QUALITY ASSURANCE AND QUALITY CONTROL

JANUARY 1992

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Demilitarization and Technology Branch
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REPORT DOCUMENTATION PAGE					
1a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED		1b. RESTRICTIVE MARKINGS None			
2a. SECURITY CLASSIFICATION AUTHORITY N/A		3. DISTRIBUTION/AVAILABILITY OF REPORT Unlimited			
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE N/A					
4. PERFORMING ORGANIZATION REPORT NUMBER(S)		5. MONITORING ORGANIZATION REPORT NUMBER(S)			
6a. NAME OF PERFORMING ORGANIZATION - Andrus Research Corporation Salt Lake City Office		6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION STEDP-MT-TM-A Dugway Proving Ground		
6c. ADDRESS (City, State, and ZIP Code) 4600 East-West Highway, Suite 900 Bethesda, MD 20814		7b. ADDRESS (City, State, and ZIP Code) Dugway, Utah 84022-5000			
8a. NAME OF FUNDING/SPONSORING ORGANIZATION U.S. Army Armament, Munitions and Chemical Command		8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER Contract DAAD09-87-D-0008		
8c. ADDRESS (City, State, and ZIP Code) Maintenance Management Division, Demilitarization and Technology Branch Rock Island, Illinois 61299-6000		10. SOURCE OF FUNDING NUMBERS			
		PROGRAM ELEMENT NO	PROJECT NO	TASK NO	WORK UNIT ACCESSION NO
11. TITLE (Include Security Classification) Development of Methodology and Technology for Identifying and Quantifying Emission Products from Open Burning and Open Detonation Thermal Treatment Methods. Field Test Series A, B, and C - Volume 2.					
12. PERSONAL AUTHOR(S) Mr. MacDonald Johnson					
13a. TYPE OF REPORT Final Report	13b. TIME COVERED FROM 12-88 TO 1-92		14. DATE OF REPORT (Year, Month, Day) 92 January		15. PAGE COUNT
16. SUPPLEMENTARY NOTATION					
17. COSATI CODES		18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number) open burning; open detonation; OB/OD; TNT; double base propellant; manufacturers residue propellant; air emissions; thermal treatment; carbon balance; emission factor; (Cont'd on reverse)			
19. ABSTRACT (Continue on reverse if necessary and identify by block number) The report describes the quality assurance/quality control program conducted during 1990 field testing which supported the OB/OD Thermal Treatment Methods Study. The QA/QC program encompassed sample collection, preparation, storage, extraction, analytical instrument operation, data reduction, statistical data analyses and interpretation. Samples included atmosphere collected in evacuated stainless steel canisters, soil, and Teflon™-coated-glass-fiber media. The supercritical fluid chromatograph and gas chromatograph with mass spectrometers were the principal laboratory analytical instruments used during these field tests. Real-time instruments detected and quantified CO, CO ₂ , NO, NO ₂ , and NO _x . Near-real-time analyses were accomplished by use of a Teflon™ bag in which samples of the plume were collected for analyses during testing operations. Sample-tracking was conducted using a system which permitted precise identification of individual specimens from collection through analyses or archiving. Analysis of atmosphere and soil samples spiked by the U.S. Environmental Protection Agency (EPA) reflected a degree of accuracy well within acceptable limits. (Continued on Reverse)					
20. DISTRIBUTION/AVAILABILITY OF ABSTRACT <input type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT <input type="checkbox"/> DTIC USERS			21. ABSTRACT SECURITY CLASSIFICATION UNCLASSIFIED		
22a. NAME OF RESPONSIBLE INDIVIDUAL Mr. MacDonald Johnson			22b. TELEPHONE (Include Area Code) (309) 782-3980/5534		22c. OFFICE SYMBOL AMSMC-DSM-D

18. (Cont'd)

supercritical fluid chromatography; SFC; demilitarization; munition dispersal; explosive dispersal; environment; BB; air building; U.S. Environmental Protection Agency; EPA; quality assurance; QA; quality control; QC; RDX; composition B; explosive D; M1 propellant; M6 propellant; single-base propellant; propellant manufacturing residue; air emissions; soil contamination.

19. (Cont'd)

Included in this report are the quality assurance program plan, test design plan, QA auditor reports, and reports of EPA-conducted audits. Almost all findings were highly satisfactory; most of those that suggested corrective action were minor and immediately resolved on-site.

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FOREWORD

A test planning directive to conduct the OB/OD test in support of U.S. Army Armament, Munitions and Chemical Command (AMCCOM) was issued by U.S. Army Test and Evaluation Command (TECOM) on 28 April 1988¹. A Technical Steering Committee Symposium was convened in July 1988. The requirement for identifying and quantifying emissions from the open detonation of explosives and open burning of propellants was discussed in detail by authorities from throughout the military, academic, and commercial communities. Conclusions and recommendations developed during the symposium are reported in proceedings of the symposium². A series of TNT detonations and propellant burns were characterized in a BangBox (chamber) in December 1988 and January 1989 for the purpose of developing methodology and technology for large scale detonations and burns in the field. The field tests took place in 1989 and 1990 and are reported in two volumes.

Volume 1. A summary which describes the planning phase, the conduct of trials, sample analyses and results, and the conclusions and recommendations. It is useful for those who need only a quick review (executive summary) and those who need a detailed description of the conduct and results of the Field Tests Phases A, B, and C.

Volume 2, Part A. A stand-alone document which covers the quality assurance and quality control procedures, the blind spiking of samples, the on site challenges of equipment and personnel, the conclusions, and the recommendations.

Volume 2, Part B. The quality assurance (QA) program plan which was developed specifically to support phase "C" field testing. While directed to phase "C" testing, it also represents the procedures and techniques and QA philosophies which were used during OB/OD field testing phases "A" and "B" and is based on experience gained during these two earlier field tests.

¹Letter, AMSTE-TA-F, Headquarters, U.S. Army Test and Evaluation Command, Aberdeen Proving Ground, Maryland, 20 April 1988, subject: Test Planning Directive for Special Study of Open Burning/Open Detonation (OB/OD), Phase II, TECOM Project No 2-CO-210-000-017.

²*Proceedings of the Technical Steering Committee Symposium 6-8 July 1988*, Headquarters, United States Army Armament, Munitions and Chemical Command, Rock Island, Illinois, August 1991.

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¹Appendices A, B, C. and D are in Volume 2 B.

SECTION 1. BACKGROUND

1.1. Program

1.1.1. Study Requirement

The amount of obsolete and/or unsafe propellants, explosive materials, and pyrotechnics (PEP) awaiting treatment is conservatively estimated to be about 200,000 tons. Historically, open burning (OB) and open detonation (OD) have been the treatment methods of choice because of their cost-effectiveness, safety, and speed. As environmental legislation has become more restrictive over government and commercial activities, OB/OD procedures have faced increasing challenges from Federal and state environmental regulatory agencies. The OB/OD Thermal Treatment Emission Study is designed to gather information on the impact of these methods on the environment.

1.1.2. Objective

This OB/OD report details the initial efforts in gathering data necessary for permitting, i.e., the development and validation of methods and procedures for collecting and analyzing emissions resulting from OB/OD operations. This has been accomplished through a four-phase test program: the BangBox (BB) Test and field test Phases A, B, and C described in separate volumes of this report.

1.1.3. Test Time Period

The test schedule is presented in Table 1.1.

Table 1.1 Open Burning/Open Detonation Test Time Period.

Test Phase	Period of Testing
BangBox Tests	7 Dec 1988 - 16 Feb 1989
Phase A Tests	13 Jun 1989 - 21 Jun 1989
Phase B Tests	16 Oct 1989 - 1 Nov 1989
Phase C Tests	7 Aug 1990 - 18 Sep 1990

1.1.4. BangBox Test

The BB Test involved the detonation of 0.5-lb amounts of trinitrotoluene (TNT) and the burning of 1-lb amounts of propellants in an air chamber approximately 1000 m³. By suspending the TNT charges above metal plates and by burning the propellant in metal pans, no soil was introduced into the resulting cloud of emission products. This test was designed to evaluate the various chemical and physical measurement systems that were being considered for use aboard the sampling aircraft in the field tests (phases A, B, and C) and to validate the carbon balance method of estimating emission factors (EF) for selected combustion products of detonation/burning.

1.1.5. Field Test Phase A

During phase A, a limited number of 2000-lb TNT detonations and 3000-lb M30 propellant burns were conducted. Immediately following each detonation or burn, samples of combustion products were collected and particles were measured by instruments aboard an aircraft which passed through the resulting cloud of gaseous products. In these tests, the TNT was contained in thin-walled metal cylinders set on the surface. The propellants were burned in metal pans. The phase A tests were designed to correlate the results of the deflagration of the small amounts in the BB test chamber with those of the much larger amounts in the field.

1.1.6. Field Test Phase A

During phase B, the field tests of TNT were expanded to include the detonation of similar amounts in steel cylinders suspended approximately 40 feet above the surface and to burn an additional type of propellant, manufacturer's residue. In this manner, phase B permitted a comparison of emissions from the surface and suspended detonation of TNT to the BB test and to each other.

1.1.7. Field Test Phase C

Phase C, the most comprehensive of the field tests, involved the surface detonation of explosive D, RDX, compound B, and TNT, and the burning of several additional types of propellants M1, M6,

and manufacturing residue. A limited number of small-scale tests of the new materials were conducted in the BB test chamber to develop target analyte lists for the new test materials.

1.1.8. Scope of Field Testing

Phases A, B, and C introduced sampling soil and fallout material and sampling both detonation- and burn-produced plumes with an instrumented aircraft. All field testing required chemical analyses of samples for targeted organic compounds and elemental materials.

1.1.9. Scope of Quality Assurance Report

This volume summarizes the quality assurance activities and accomplishments of BB, phase A, phase B, and phase C testing. Previous reports provide considerable detail on the quality assurance support of BB and phase A testing (References 1 and 2, Appendix C), there will be some repetition of information in this report. This QA report emphasizes the BB test (during which the sampling and measurement/analysis systems were developed and optimized) and phase C (during which the most extensive field testing was conducted).

1.2. QA Background

1.2.1. Test Planning

The OB/OD tests, because of their complexity and potential implications, required extensive planning to ensure validity, reliability, and repeatability. The test design included an adequate number of replicate tests to accommodate statistical testing and to support estimating the inherent variability of the test/measurement systems.

1.2.2. Documentation

Procedures and conditions of the test and the measurement systems were recorded and are reported. Letters of instruction (LOIs), discussed below, were used in lieu of standard operating procedures (SOP) for process documentation.

1.2.3. Technical Steering Committee

The overall planning for the scientific investigation was accomplished by a technical steering committee (TSC) organized by, and accountable to, the PM. The TSC consisted of consultants and distinguished authorities drawn from industry, government agencies, and academia. Composition of the TSC is defined in the Acknowledgement Section of Volume 1 in this report.

1.2.4. Letters of Instruction

Letters of instruction (LOIs) provided a greater degree of flexibility than is possible with standard operating procedures (SOP's). Any changes to LOI procedures, test conditions or measurements/analyses were recorded and the appropriate LOI revised to properly document the change. All LOI's were prepared by the respective operational organizations and their internal quality assurance personnel and then subjected to review by the TSC, OB/OD technical director, the OB/OD QA agency (Environmental Labs Inc), and the U.S. Environmental Protection Agency (EPA). The LOI's were an important part of the QA Project Plan (QAPP). Those LOI's used in phase C are included in this report (Volume2, Part B, Appendix C). Several other LOI's used in earlier phases are also included in this same volume.

1.2.5. Quality Assurance Project Plan

The QAPP for each phase was prepared by Environmental Laboratories, Inc. (ELI), to define all of the planned activities for the internal QA systems of the operating organizations and the external quality systems audits by ELI. The QAPP documented the intended systems for the four major stages of the QA cycle, i.e., planning, execution, appraisal, and corrective action.

1.2.6. Short-Term and Long-Term Appraisal and Corrective Actions

The short-term appraisals and corrective actions were handled on an ongoing basis by the operational groups or laboratories. Longer-term or major appraisals were conducted and corrective actions initiated by either the TSC, ELI, or EPA. Dr. Booth of ELI and Dr. Mitchell of EPA, both members of the TSC, enhanced coordination between technical and quality assurance.

1.2.7. Plans for Replications

In the planning of test replications, considerations were given to (1) the basic type of test, (2) the materials used in the tests, and (3) the conditions under which the tests were performed. The plans by the TSC for adequate replication of tests to achieve scientific validity were also paralleled by plans for adequate replication of samples and replication of measurements/analyses for statistical and QA purposes. The plans for replication of samples and trials were generally made jointly by the TSC and QA personnel and were included in the test design plan. The requirements for replicate measurements or analysis were part of the QAPP and/or the LOI's.

1.2.8. Aspects of Quality Assurance

In general, the QAPP covered two main areas of activities: those within the operational groups (internal QC) and those external to the operations groups(external QC or QA. All of these activities, whether internal or external, are considered as QA within the OB/OD context.

1.2.9. Systems Audits

Systems audits involved visits by QA personnel to the operational areas, typically during a period in which the audited activity was actively engaged in OB/OD activities, to evaluate the activity's internal QC system on a qualitative basis.

1.2.10. Performance Audits

The performance audits consisted of challenging the measurement/analysis systems of the operational groups with carefully and accurately prepared blind samples, or unknowns. The results, in quantitative terms, were used to evaluate the accuracy of the measurement systems. In some cases, estimates of the precision of the measurement systems could be made from the results of the performance audits. Estimates of precision were also made from the internal quality control data. The audit activities by ELI and EPA are discussed more fully in Sections 2 and 3.

1.2.11. Overlap of QA and Technical Considerations

Technical considerations and QA considerations were difficult to completely separate, because they critically overlapped in some aspects of the project. Members of the TSC were keenly aware of the need for the most meaningful data of highest quality attainable under the constraints of state-of-the-art capability and project funding. As members of the TSC, Dr. Gary Booth, the QA Director for OB/OD, and Dr. William J. Mitchell, Associate Chief of EPA's Atmospheric Research and Exposure Assessment Laboratory (AREAL), shared this perception.

SECTION 2. ORGANIZATIONS PROVIDING QUALITY ASSURANCE

2.1. Key Personnel

The following table lists key personnel supporting the OB/OD study during the BB test and field testing phases A, B, and C. The table indicates the continuity, throughout the study, of the organizations and individuals involved. The high degree of continuity eliminated the need for training or orientation of new personnel, thus improving the efficiency and effectiveness of the effort and enhancing compliance to the various procedural and QA requirements. The table is also of value by identifying individuals holding QA responsibilities.

Table 2.1a Key Personnel Holding Quality Assurance Responsibilities.

ORGANIZATION	INDIVIDUAL	BB	A	B	C
Alpine West Laboratories	Dr. Milton Lee Dr. Karin Markedis Dr. Christine Rouse Mr. Michael Dee Dr. Edgar Lee Mr. Wayne Lee Mr. Bill Vorkink	X QA X X	X QA X X	X QA X	X X X
Environmental Labs Incorporated	Dr. Gary Booth Mr. Floyd McMullin, Jr Mr. Todd Parrish Mr. Corbin Coombs Mr. R.C. Rhodes	QA QA QA QA	QA QA QA QA	QA QA QA	QA QA QA
U.S. Environmental Protection Agency	Mr. Chester Oszman Dr. William Mitchell Mr. R.C. Rhodes Mr. William Barnard Ms. Linda Porter Mr. Howard Crist Ms. Avis Hines Ms. Elizabeth Hunike Ms. Lisa Smith Ms. Ellen Streib Mr. Jack Bowen Mr. Oscar (Bud) Dowler	X QA QA QA QA QA QA QA QA QA QA QA QA	X QA QA QA QA QA QA QA QA QA QA QA	X QA QA QA QA QA QA QA QA QA QA QA	X QA QA QA QA QA QA QA QA QA QA QA

Table 2.1b Key Personnel Holding Quality Assurance Responsibilities (Cont'd).

ORGANIZATION	INDIVIDUAL	BB	A	B	C
Battelle Columbus Division	Dr. Laurence Slivon Ms. Jean Czuzwa Mr. James Chuang Mr. Mark Bower Mr. Denise Contos Mr. Dave Oiler Mr. Ramona Mayer	QA X X X X X QA			
Oregon Graduate Institute of Science & Technology	Dr. Reinhold Rasmussen Mr. Robert Daluge Mr. Don Stern Mr. Bob Watkins	QA X X X	QA X X X	QA X X X	QA X X X
Lawrence Berkeley Laboratory	Mr. Robert D. Giauque Mr. Joseph M. Jaklevic Ms. Linda Sindelar	QA X	QA X X	X	QA X X
Sunset Laboratories	Mr. Robert Cary	QA	QA	QA	QA
Lockheed Engineering and Sciences Company	Mr. LaVon Stokes Mr. Jim Stephens Mr. Lamont Law	X X QA	X X QA	X X QA	X X QA
Sandia National Laboratories	Mr. Wayne Einfeld Mr. Brian V. Mokler Mr. Monty Apple Mr. Dennis Morrison	QA X X	QA X X	QA X X	
U.S. Naval Ordnance Station, Indian Head	Mr. Daniel LaFleur Mr. Randy Waskul	X	X	X	X X
U.S. Army Dugway Proving Ground	COL Jan Van Pruyen COL Frank Cox COL Wyette Colclasure III Mr. Kenneth Jones Mr. John Woffinden Mr. James Bowers CPT David Coxson CPT Kevin Janes	X X X X X X X	X X X X X X X	X X X X X X X	X X X X X X X
Sunset Laboratory	Mr. Robert Cary	X	X	X	

Table 2.1c Key Personnel Holding Quality Assurance Responsibilities (Cont'd).

ORGANIZATION	INDIVIDUAL	BB	A	B	C
Andrulis Research Corporation	Dr. Ray Bills Dr. Kenneth Zahn Mr. Cecil Eckard Mr. Douglass Bacon Mr. Duane Long Mr. A. Lacy Hancock	X X X X X X	X X X X X X	X X X X X X	X X X X X X
U.S. Army Armament, Munitions and Chemical Command	Mr. Dean Sevey Mr. MacDonald Johnson	X X	X X	X X	X X
Consultants	Dr. H. Smith Broadbent Dr. Dale Richards Dr. Nolan Mangelson Dr. Randy Seeker Mr. Wayne Ursenbach	X X X X X	X X X X X	X X X X X	X X X X X

2.2. Internal Quality Assurance

All personnel actively expressed their commitment to quality. During field testing and laboratory analysis, personnel working on OB/OD tasks complied with procedural and QA requirements. The majority of supporting organizations had a designated QC specialist involved in QA planning before OB/OD testing began. These specialists held oversight and quality evaluation authority during the conduct of the project. In the smaller organizations, the QA specialist commonly held other responsibilities, but was able to devote sufficient attention to the QA role to ensure compliance with internal procedures.

2.2.1. Sunset Laboratory

Sunset Laboratory (SSL) typifies the smaller-scale operation which competently adhered to QC requirements. Mr. Robert Cary is the owner and also the supervisor, laboratory chemist, and quality assurance specialist. Quality assurance audit results confirmed that SSL consistently maintained a fully satisfactory QC program.

2.2.2. Oregon Graduate Center

The Oregon Graduate Center (OGC), now titled the Oregon Graduate Institute of Science & Technology, also supported OB/OD analyses with a relatively small laboratory. Dr. Reinhold Rasmussen is both the project manager and director of quality assurance. An internationally recognized expert in his field, Dr. Rasmussen maintained a very acceptable quality performance program within his laboratory while directing complex chemical analyses.

2.2.3. Lawrence-Berkeley Laboratory

At Lawrence-Berkeley Laboratory (LBL), Mr. Robert D. Giauque is responsible for the sample handling and data analysis for x-ray fluorescence (XRF) analysis; thus, in effect, he is responsible for QA at LBL. Mr. Joseph M. Jaklevic is the Project Manager.

2.2.4. Battelle-Columbus Division

Battelle-Columbus Division has an internal organizational QA program that extends across projects. Mr. Ramona Mayer, of the general BCD staff, acts as the QA advisor for the OB/OD activity. Dr. Lawrence Slivon, as the project manager for Battelle's OB/OD analytical support, assumed significant responsibility for the quality of BCD analytical results.

2.2.5. Alpine West Laboratories

Dr. Karin Markedis directed QA activities at AWL until her departure. At that time, Mr. Bill Vorkink was assigned QA responsibilities.

2.2.6. Sandia National Laboratories

Because very few individuals at SNL were directly involved in the OB/OD study, Mr. Wayne Einfeld served as both project manager and QA director for SNL support of the OB/OD study.

2.2.7. Lockheed Engineering and Sciences Company

A relatively large group of LESC workers was involved in the field sampling of soil, fallout, and burn residue and in the handling, weighing, and compositing of these samples. Mr. Lamont Law was appointed director of LESC OB/OD quality assurance, and Mr. Jim Stephens was designated supervisor of the field sampling and sample handling activities. During phase C, Mr. Stephens was drawn away from the OB/OD project and Mr. Law assumed his responsibilities.

2.2.8. General

Because the organizations discussed above had various assignments of QA responsibility, the effectiveness of individual internal QA/QC program depended on the capability and initiative of the QA supervisory personnel and the support of the organizational project manager. Overall, the internal QA/QC programs were highly satisfactory.

2.3. Internal Quality Control

The internal QC systems addressing field activities were somewhat different for the BB Test (conducted in a large test chamber) than for field testing phases A, B, and C (conducted in open desert terrain at DPG). One of the salient features of the BB test chamber (an inflatable hemisphere) was that the combustion products from burning propellant or detonated explosive are contained in the chamber for a lengthy time (almost indefinitely), except for the dilution effect of the inflation fan and very minimal leakage to the outside atmosphere through small holes in the fabric. Sampling inside the chamber was continued for a considerably longer time than was possible when using an aircraft which could sample a cloud for only a few seconds at a time. Another basic difference was that there was no soil or fallout to sample in the enclosed test chamber, whereas collecting both of these types of specimen were important considerations during field testing. Laboratory analyses were essentially the same, except for the amounts of sampled material.

2.3.1. BangBox Test

2.3.1.1. Documentation and Data Management

Special precautions were taken throughout all OB/OD activities to ensure the integrity of samples and resulting analyses. Logbooks and journals were maintained, and subject to QA audit, the sample-tracking system (described below) provided a "cradle-to-grave" record of sample handling. In some instances, computers backed up journal entries. The QA agency maintained a central repository for OB/OD records of importance.

2.3.1.2. Sample Tracking

2.3.1.2.1 A "chain-of-custody" sample-tracking scheme, such as is used to provide for proper handling of evidence in legal cases to provide assurance that sample identification is maintained, was used throughout the OB/OD study. This system used the sample custody forms, receipt forms, and storage forms shown in Volume 2, Part B, Appendix B of this report. Identification of every sample was maintained from the initial collection through the analysis and data processing. A record that correlated identification numbers with a description of the corresponding sample was maintained to assist in sample and data management. The lists were updated as the need for changes became evident, such as when samples were split or errors in a description for a given number were discovered.

2.3.1.2.2 The QA agency (ELI) monitored testing to ensure that sample custody forms were prepared for all samples taken during testing. The sample forms were somewhat revised for phases A, B, and C to make them more appropriate for specific sample-handling situations.

2.3.1.2.3 Laboratories which assigned their own internal sample numbers to samples entering the laboratory maintained an accurate cross-reference listing between the OB/OD sample numbers and the laboratory internal numbers.

a. AWL and BCD used "in-house" tracking forms, but the remaining laboratories used alternative schemes. At LBL, samples were logged in, given a test sequence number, and cross-checked both manually and by computer to ensure accuracy. At SNL, the sample number and the date were logged into a notebook. At OGC, numbers were assigned to each cylinder and entered into a logbook prior to being sent to the BB test site. When the cylinders were returned to OGC for analysis, dates were logged and numbers were checked for accountability. As part of the

analytical routine, OGC also cleaned their cylinders immediately after analysis. These alternative, internal sample tracking systems were considered effective.

- b. During the BB test, a sample was split for analysis or archiving reasons, the collection report was copied so that the duplicate form could accompany the additional sample. This created a potential for confusion when tracking samples or compiling results, since one distinct portion of the same sample carried an original form and the other a copy of the same form. In phases A, B, and C of the OB/OD study, a new number was assigned to split or composite samples, and the original source sample number was referenced.
- c. SNL personnel did not use receipt, custody, and storage forms to track the TNT blocks used in the BB detonations because the facility employed an alternative internal tracking procedure which ELI judged satisfactory. ELI received copies of the original shipping documents, and collection reports were used for the samples of the TNT blocks that were taken for elemental (C,H,N) or trace analysis.
- d. Sample custody forms were completed on both the soil and resin samples submitted to EPA for spiking and for use as audit samples. Collection and shipping forms were not employed when handling these sets of samples. Although the recommended forms were not employed in all cases, ELI judged that no permanent effect on proper data identification or quality ensued.

2.3.1.3. Laboratory Logbooks

No specific format was required to be used by the various active organizations, since each organization had devised and maintained their own internal system for a considerable period of time.

2.3.1.3.1 The specific system used in each laboratory was reviewed during systems audits by QA personnel and are described below. These systems were maintained throughout the OB/OD study for the BB Test and subsequent phases A, B, and C. Most supporting laboratories (AWL, BCD, LBL, and SSL) used sample custody numbers when logging in samples. A few assigned a unique internal laboratory identification number to each sample received: OGC used the numbers they

had assigned to outgoing tanks, and SNL used a standard institution-wide sample numbering scheme.

a. Alpine West Laboratories

AWL maintained a project-specific sample logbook for recording sample receipt. The supercritical fluid chromatograph-mass spectrometer (SFC/MS) operator used a project-specific personal logbook to record extraction information, all samples injected, and details of instrument performance. Because the SFC/MS was not used for any other project, no additional instrument logbook was maintained, but all calibration printouts for the SFC/MS were put in a dedicated looseleaf notebook. The temperature of the AWL freezer was reportedly checked monthly, but there was no documentation.

b. Battelle-Columbus Division

BCD recorded each sample receipt on a separate line in a logbook, using the sample custody Number and sample description. The book number, line number, and sample custody number became the "in-house" sample identification number. A form was used at the freezer to record in-out times and volume sampled, according to identification number. Up-to-date temperature-monitoring sheets were located at the freezer. Each worker maintained a project-specific personal logbook to enter data concerning the sample taken, the time, the volume injected into the gas chromatograph-mass spectrometer (GC/MS), etc. A logbook was used at the GC/MS for recording samples analyzed, analyst name, and instrument-related details.

c. Lawrence-Berkeley Laboratory

LBL employed a large logbook to record sample identification, instrument performance, QC results, and location of corresponding experimental data. Removable hard disks were used to store all experimental data, including sample numbers and calibration information. The logbook was written in pencil to allow neat corrections, but data integrity was not threatened, because all data was stored electronically.

d. Oregon Graduate Center

OGC used personal logbooks to record tank identification and pertinent analytical information. Calibration information was stored with the corresponding analytical data. Accounting was not needed for long-term sample storage, because tanks were cleaned immediately after analysis.

e. Sandia National Laboratories

Project personnel at SNL used personal logbooks to record sample numbers, timing information, etc. A project-specific logbook was maintained for each individual instrument for recording information such as zero and span checks, calibration, and maintenance. Data from the individual notebooks was also recorded electronically, and real-time instruments directly produced electronically-recorded signals.

f. Sunset Laboratory

The principal investigator used a personal logbook to record all information concerning analysis runs. In addition, the instrument calibration and analysis printout was stored electronically.

2.3.1.4. Written Procedures

2.3.1.4.1 Letters of Instructions

a. The investigative nature and scheduling of the BB test made it impractical for the supporting activities to develop formal standing operating procedures (SOPs) in advance. As is common in such situations, letters of instruction (LOIs) were employed to accommodate approved changes in procedure during methodology development. The TSC was required to approve the proposed changes before they could be implemented and the LOI's revised. The principal investigators documented or confirmed all procedures described in the LOIs to ensure that the LOIs adequately described the methodologies being used. The development, application, or supervision of procedure by nationally and internationally recognized authorities was instrumental in ensuring achievement of high quality.

b. After suggested revisions were considered and incorporated, a copy of the revised LOI was filed with ELI, which functioned as a clearinghouse, archiving agency, and auditor.

2.3.1.4.2 BB Test Letters of Instruction

Listings of the LOI's for the BB test can be found in the BB report Volume 3, Appendix B.

a. Alpine West Laboratories

At the time of the site visit (25 Jan 1989), AWL used acetonitrile to extract the filter samples, in accordance with the LOI. On 4 February 1989, the LOI was revised and methylene chloride was used to extract samples taken during the 31 January through 16 February 1989 trials. At the time of the visit, AWL workers were extracting all the samples (from preliminary BB trials) and sending one-half the extract to BCD for analysis. For the main series of BB trials, the samples analyzed at BCD were all extracted at BCD.

b. Battelle-Columbus Division

As specified in the LOI, a calibration standard was analyzed daily (noted by the auditor during the site visit on 28 December 1988). For the 21 January through 16 February series of trials, BCD used a moving average response factor based on the analysis of standards that bracketed, in time, a particular group of samples. All CI-SIM results were based on the new procedure, documented as BCD LOI dated 28 May 1989.

c. Lawrence-Berkeley Laboratory

LBL did not produce a LOI specifically for the BB analyses, but provided a generic written description of their EPA-approved procedure. The analyses were conducted according to a well-practiced routine, and no deviations from the written procedures were noted other than, at the time of the visit (31 Jan. 1989), data were being recorded on a 20-mByte Bernoulli disk instead of a 5-mByte disk.

d. Oregon Graduate Center

Because OGC had not provided a LOI by the time of the site visit (24 Jan. 1989), the audit focused on compliance with good laboratory procedures (GLP). The procedures employed by the laboratory personnel appeared to be meticulously done.

e. Sandia National Laboratories

At the beginning of the BB trials, SNL provided an LOI that dealt with the real-time instruments. During the ELI site visit on 17 December 1987, these instruments were not being used. However, the EPA performance and systems audit on 6 to 8 February 1989 included real-time instruments. The analysis of HCN and NH₃ bubblers was observed by ELI; an LOI was not prepared because a published procedure was followed. SNL appeared to comply with good laboratory procedures.

f. Sunset Laboratory

The analyst routinely performed a very specialized, EPA-approved procedure developed at SSL. The LOI accurately described his manipulations.

2.3.1.5. Data Management and Archival

2.3.1.5.1 The QAPP required that all data be submitted to ELI for QA review, reproduction, and archiving (originals), before copies were distributed for data reduction and analysis. However, as the project progressed, certain key data were forwarded (principally by SNL) directly to ANDRULIS Research Corporation (ARC), prior to being received by ELI, to expedite the data reduction process. The QAPP also stipulated that ELI check the data for quality and completeness before distribution. While ELI's staff did check the data for general adherence to proper record-keeping practices, technical examination of each item of data by ELI was not feasible, because of the quantity of data involved and since defects in individual datum often only become apparent as detailed calculations are performed on the whole data set. Consequently, ARC's scientific staff also functioned as technical data quality validators as data were reduced and analyzed. Discrepancies so identified were brought to the attention of ELI and referred to the involved laboratories for

resolution. The responsibilities for receiving and distributing technical reports, including reports of sample analyses, were changed for phase C (See Section 2.3.2.1.2.).

2.3.1.6. Sampling and Real-Time Measurements

2.3.1.6.1 Sampling Accuracy

- a. Samples containing the OB/OD detonation and combustion products were carefully and properly handled, so that results from subsequent sensitive analytical methods would not be jeopardized. Since one purpose of the BB trials was to evaluate candidate sampling methods for potential use in future studies, checking and comparing sampling systems and results occupied a large proportion of the BB field and data analysis efforts.
- b. The BB test chamber is an air-supported, rubber-coated fabric hemisphere with a radius of 7.6 meters. A 5.5-meter-long x 2.1-meter-high x 2.5-meter-wide plywood airlock provides access to the building. Air pressure supplied by a blower supports the building. A number of sampling instruments normally installed on the SNL atmospheric research aircraft (which were used in later, outdoor large-scale OB/OD tests) were positioned both in the airlock and inside the chamber so that data derived from direct chamber air and indirect (tube- or probe-sampled) chamber air could be compared. The comparison allowed determining if inaccuracies were introduced by the 5-meter-long, 8-cm-diameter aluminum tube that would subsequently serve as the aircraft sampling probe for particulate and gas samples during field testing.
- c. Table 2.2 lists samplers and real-time instruments used in the BB airlock. Air from the aircraft sampling probe (which extended from the airlock into the BB) was routed through a pneumatically driven 10-cm-diameter gate valve into a 1.5-m³ carbon-impregnated polyethylene (Velostat™) sampling bag. The bag, constructed of electrically-conductive plastic material in order to minimize wall loss of charged particles, filled with air from the chamber interior in approximately 40 seconds. Stainless steel sampling lines connected to the aircraft probe led to filters, vapor collection systems, and real-time gas monitors also located in the airlock.

Table 2.2 Samplers and Real-Time Continuous Monitors Located in the BangBox Airlock.

Number ^a	Instrument	Purpose	Model
1	CO ₂ gas analyzer	RTC ^b	TECO 41H
2	CO gas analyzer	RTC	TECO 48
3	SO ₂ gas analyzer	RTC	TECO 43
4	O ₃ gas analyzer	RTC	TECO 49
5	NO _x gas analyzer ^c	RTC	CSI 1600
6	THC analyzer (FID)	RTC	Century OVA-108
7	THC analyzer (PID)	RTC	HNU Model PI-101
8	Differential mobility particle spectrometer	Particle sizes 0.01 to 0.5μm	TSI DMPS
9	Aerodynamic particle spectrometer	Particle sizes 0.5 to 15μm	TSI APS
10	Teflon™ filter sampler	Metals analysis	GAST Model 30
11	Polycarbonate filter sampler	Particle morphology	GAST Model 30
12	semi-VOST sampler	Filter and resin samples of semivolatile organics	GMW PS-1
13	6-L Stainless steel evacuated canisters (VOC)	Chamber atmosphere	OGC custom design
14	0.85-L Stainless steel (SF ₆) evacuated canister	Chamber volume	OGC custom design

^aItems 1-12: See SNL LOI, Gas and Aerosol Instrument Calibration and Sampling Procedures.

Items 13-14: See OGC LOI, VOC Collection Analysis System.

^bReal-time concentration.

^cAlso analyzes for NO and NO₂, by difference.

d. Particulates and semivolatile organic compounds were collected by two semivolatile organic sampling trains (semi-VOST), each consisting of a modified commercially-available sampling unit containing a prefired quartz filter followed by two cartridges containing XAD-2™ resin. (Preliminary trials eliminated Porapak R™ resin because its flattened resin particles are subject to airflow-restricting packing.) The leading XAD-2™ cartridge in the semi-VOST, containing 65 grams of resin, was backed up by a cartridge containing 20 grams of resin. The second cartridge was included to recover any component that might pass through the first cartridge during aspiration.

e. Other filters connected to the bag outlet manifold included a Teflon™ filter used for gravimetric analysis (for particulates) and XRF measurements (for elements), and a Nuclepore™ polycarbonate filter used for scanning electron microscopy (SEM) (to examine particle morphology). Five carbon-vane pumps that supplied a total airflow of approximately 200 liters per minute provided air movement from the bag through the filter. Mass flow meters enabled calculating total airflows through the samplers. In-line Teflon™ filters were used with real-time instruments to prevent contamination of the instrument optics by particulates. Air input to these instruments was selected by a manual valve from either the main sampling probe or from the sampling bag. A differential mobility particle sizer to measure particle size distributions in the 0.01- to 0.5- μm particle diameter range. On selected test days, a continuous flame ionization detector and a photoionization detector provided an approximate measure of volatile hydrocarbon concentrations in near real-time.

f. To determine the magnitude of sorption on the inside surface of the bag, or the extent of offgassing from the bag material, OGC personnel collected grab-samples of air in electropolished, passivated 6-L evacuated stainless steel cylinders directly from the sampling duct and indirectly from the 1.5-m³ sampling bag. OGC subsequently assayed the contents of these cylinders for H₂, CO, CO₂ and C₁ - C₁₀ volatile hydrocarbon concentrations by gas chromatography (GC) with thermal conductivity flame ionization (for tracer SF₆ analysis), and electron capture detectors. Samples collected and analyzed from the bag, which was connected to the aircraft sampling probe, were called "indirect" samples.

g. Table 2.3 lists the instrument systems used within the BB to collect so-called "direct" samples. Two laser-particle spectrometers, normally installed and flown on the SNL aircraft, were used to make particulate measurements in real-time. The FSSP probe incorporates true in-situ measurement principles and requires no correction for particle transmission or sampling losses. Both the FSSP and ASASP probes provide records of total particle counts in 1-minute intervals. A flash-lamp integrating nephelometer and a portable forward light-scattering particulate detector provided continuous measurement of particulate concentration inside the chamber during each test. Video cameras, recordings, and (on occasion) a high-speed (5,000 frames/s) camera provided photographic coverage of detonation and burn trials.

Table 2.3 Samplers and Real-Time Continuous Monitors Located Inside the BangBox Chamber.

Number ^a	Instrument	Purpose	Model
1	Aerosol spectrometer	Particle-size distribution (0.1-3 μm)	PMS ASASP-100-x
2	Aerosol spectrometer	Particle-size distribution (1-47 μm)	PMS FSSP-100-x
3	Integrating nephelometer	Particulate concentration	MRI 1550
4	Nephelometer	Particulate concentration	MIE RAM-1
5	Video camera	Photometric record	Sony
6	Fast-frame camera (5000 frames/s)	Photometric record	Unknown
7	semi-VOST sampler	Filter and resin samples of semivolatile organics	GMW PS-1
8	Glass impinger (bubbler) ^c	HCN, NH ₃ , and HCl concentration	Gillian 113FS
9	Evacuated stainless steel tanks ^d	Volatile and semivolatile organics	OGC custom design

^a Items 1-4. See SNL LOI, Gas and Aerosol Instrument Calibration and Sampling Procedures.
 Items 5-6. Furnished through SNL Photo Documentation Division.

Items 7-8. See SNL LOI, Gas and Aerosol Instrument Calibration and Sampling Procedures.
 Item 9. See OGC LOI, VOC Collection Analysis System.

^b Two samplers were used routinely. For composite propellant burn, an additional two samplers were used.

^c Two bubblers were used, except for composite propellant burn, when two additional bubblers (in series) were used to measure HCl.

^d Nine tanks were used, except for test of 15 Feb. 89, when 27 tanks were used including three cryogenic tanks immersed in liquid N₂.

h. Two 1-meter diameter fans with approximate airflow rates of 250 m³/minute rapidly mixed the contents of the building prior to collecting those samples intended to be representative of well-mixed (homogeneous) chamber air. For purposes of initial data reduction, samples collected from the chamber interior immediately after detonation or burn and prior to turning on the mixing fans were considered to have come from "nonhomogeneous" air and were so identified in all data. The fan blade surfaces were also sampled to determine the amount of a given analyte that might have adhered to the blade.

- i. Semivolatile and organic particulate species were collected directly from the chamber interior by two modified semi-VOST samplers operating at flow rates of approximately 100 L/min. (Standard high-volume sampler blowers with no flow control were used as the air movers for these direct semi-VOST sampling units, but calibrated mass flow meters were installed in the lines to ascertain the exact flow rates, also dry test flow readings were taken with filters in line before sampling and after the sample was taken). The filter and cartridge units used to collect samples in these semi-VOST samplers were identical to those used in the airlock. The direct semi-VOST samplers were checked independently, using evacuated, passivated, electropolished 32-L stainless steel cylinders (during the trials of 31 January to 6 February 1989).
- j. Glass impinger bubblers filled with appropriate absorbing solutions were aspirated within the BB to sample any ammonia, hydrogen cyanide, and hydrogen chloride (for the composite propellant burn) that might be produced. From prior experience, SNL personnel knew it was necessary to use two bubblers in series to collect a valid HCl sample.
- k. During the composite propellant burn trial, medium-volume air samplers (283 L/min) drew duplicate samples on quartz fiber filters, backed up by precleaned polyurethane foam-filled cartridges for analysis for PCDDs and PCDFs (dioxins) by BCD. The analyses included determination of total hepta-, hexa-, penta-, and tetra-CDD and CDF congeners, as well as octa- and 2,3,7,8-tetra-CDD and -CDF concentrations (all various forms of dioxins).
- l. The TSC had previously estimated sample volumes required for the analytical methods to detect parts per billion (ppb) levels of combustion products. Mass-flow meters calibrated before and after the BB trials measured the volumes of BB air actually drawn through each sampler. Onsite measurements of temperature and atmospheric pressure were used to correct the observed sample volumes to the volumes at standard conditions.

2.3.1.7. Quality Control

2.3.1.7.1 Because of the breadth of the BB investigation, no single individual was designated as overall sampling coordinator. The PM, following consultation with members of the TSC, made the initial decisions about methods and equipment and maintained oversight of the sampling operations

at the BB. Primary onsite responsibility for sample collection details was borne by the PM as supported by each of the appropriate principal investigators (from SNL, OGC, and BCD), with assistance and guidance by members of the TSC (from ARC, BCD, BYU, and DPG).

2.3.1.7.2 Control of sample quality during the BB trials (to minimize systematic and random errors) was maintained primarily by using calibrated flow devices and calibrated real-time continuous samplers, by using a redundancy of samplers, and by including the most accurate sampling systems as a basis for comparison.

2.3.1.8. Completeness of Sampling Effort

Because this was an investigative effort, the number and type of samples taken and analyzed were expected to change from those originally specified in the Test Design Plan (TDP). Increases in the number of samples were often approved by the TSC, so that the option of further investigation through additional sample analyses would be possible. Similarly, the taking and storage of a sample was generally relatively inexpensive, but some analyses were very costly. Accordingly, not all samples were analyzed, especially if it could be determined, by review of preliminary data that the probability of useful information was low. When decisions were made onsite to take additional samples, sample custody forms were initiated and ELI made extra checks of logbooks and other records to ensure that all such samples were appropriately accounted for.

2.3.1.9. Chemical Analyses

2.3.1.9.1 Table 2.4 lists the analytical instruments or techniques used by the individual laboratories to analyze BB test samples. (The real-time continuous monitors were previously covered in Table 2.2.) The respective LOIs are included with the BB report, Volume 3, Appendix B.

Table 2.4 Instruments and Methods Used to Analyze BangBox Trial Samples.

Laboratory ^a	Instrument or Method	Type of Analyte	LOI ^b
AWL	Lee Scientific Model 602 SFC Finnigan MAT INCOS 50 MS Varian Model 3400 GC Finnigan MAT 8430 MS	Semivolatile organic compounds	AWL LOI 1-6
			AWL LOI 1-6, 8
BCD	Finnigan TSQ-45 GC-MS Finnigan 4500 MS	Semivolatile organic compounds	BCD LOI 1-2
LBL	X-ray Spectrometer (LBL design)	Metals and non-metals	LBL SOP
OGC	Perkin Elmer 3920 GC Carle 211 M GC Shimadzu GC - Mini 2 Trace Reduction Gas Detector RGD2	Volatile organic compounds	OGC LOI: VOC analysis system
SNL	See Table 2.3 for real-time instruments	Gases	SNL LOI
SSL	Thermal-optical instrument, SSL design	Elemental and volatizable carbon	SSL LOI

^aAWL - Alpine West Laboratories; BCD - Battelle-Columbus Division; LBL - Lawrence-Berkeley Laboratory; OGC - Oregon Graduate Center; SNL - Sandia National Laboratories; SSL - Sunset Laboratory.

^bLetter of instruction.

2.3.1.9.2 Detection Limits

Although the analytical methods were chosen to satisfy each individual requirement of the BB project based on expert knowledge of instrument capability, the analyte diversity and the investigative nature of the project dictated that the laboratories characterize the performance of their instruments and the adequacy of their procedures before commencing the analysis of BB samples. For example, it was necessary to determine the efficiency of extracting the analytes from the resin. Tables 2.5a,b,c presents and defines the detection limits for each of the analytical instruments or techniques.

Table 2.5a Detection Limits for Chemical Analyses Used on BangBox Samples.

Analyte	Detection Limits (ng/mL)				
	AWL ^a SFC-MS ^c		AWL Lower Instrument Detection Limit	BCD ^b GC-MS ^d	BCD Lower Quantification Limit
	PI-EI-SIM ^e	CI-SIM ^f			
2,4-Dinitrotoluene	17.6	0.07	S/N ^g = 3	4.0	Lowest standard used for calibration
2,6-Dinitrotoluene	19.3	0.05		4.0	
Dibenzofuran	21.2	0.31		60	
N-Nitrosodiphenylamine	196	0.12		60	
2-Aminonaphthalene	125				
2,4,6-Trinitrotoluene	20.4	0.06		4.0	
2-Nitronaphthalene	90.3			4.0	
4-Nitrophenol	93.2	0.30		33	
Benz[c]acridine	124			60	
Benz[a]anthracene	118	0.73		60	
1-Nitropyrene	91.8	1.30		4.0	
Benzo[a]pyrene	92.4			48	
Dibenz[a,h]anthracene	91.6			48	
1,6-Dinitropyrene	196			10	
Naphthalene		0.62			
1-Methylnaphthalene		0.62			
2-Methylnaphthalene		0.62			
1,3,5-Trinitrobenzene		0.12			
Biphenyl		1.15			
Phenanthrene		1.20			
Pyrene		0.83			
Phenol				30	
2-Naphthylamine				30	

^aAlpine West Laboratories.

^bBattelle-Columbus Division.

^cSupercritical Fluid Chromatography - Mass spectrometry.

^dGas Chromatography - Mass spectrometry.

^ePositive ion, electron impact, selective-ion monitoring.

^fChemical ionization, selective-ion monitoring.

^gSignal-to-noise ratio.

Table 2.6b Detection Limits for Chemical Analyses Used on BangBox Samples (Cont'd).

Analyte	LBL ^a Detection Limits (ng/cm ²)	Instrument Method	Lower Detection Limit
Aluminum	150		
Silicon	40		
Sulfur	15		
Chlorine	12		
Potassium	6		
Calcium	5		
Titanium	30		
Vanadium	20		
Chromium	15		
Manganese	12		
Iron	12		
Nickel	6		
Copper	6		
Zinc	6		
Gallium	4		
Germanium	3		
Arsenic	3		
Selenium	2		
Bromine	2		
Rubidium	2		
Strontium	3		
Lead	7		
Zirconium	8		
Molybdenum	6		
Silver	5		
Cadmium	6		
Tin	8		
Antimony	8		
Iodine	12		
Barium	35		

^aLawrence-Berkeley Laboratory.

^bX-ray fluorescence.

Table 2.5c Detection Limits for Chemical Analyses Used on BangBox Samples (Cont'd).

Analyte	SNL* Detection Limits	Units	Instrument Method	Lower Detection Limit
Carbon Dioxide	1.2	ppmv	TECO Model 41H	S/N = 2
Carbon Monoxide	0.1	ppmv	TECO Model 48	
Sulfur Dioxide	2	ppbv	TECO Model 43	
Ozone	5	ppbv	TECO Model 49	
Oxides of Nitrogen	6	ppbv	CSI Model 1600	
Total Hydrocarbons	2	ppmv	Century PVA-108	
Total Hydrocarbons	0.1	ppmv	HNU Model PI-101	
Hydrogen Cyanide	0.5	µg	CN elect.; NIOSH 116	Literature
Hydrogen Chloride	5.0	µg	Cl elect.; NIOSH 115	
Ammonia	0.3	µg	Colorim.; NIOSH 205	
Sunset Laboratory detection limits				
Organic/elemental carbon speciation	0.3	µg/cm ²	Thermal optical instrument	3 Standard deviations of repeated readings of blank

*Sandia National Laboratories.

a. Alpine West Laboratory

An instrument "limit of detection" was defined as the compound concentration that would give a signal/noise ratio of 3, when only the pure individual compound is dissolved in the solvent used for extraction. (This common index of the ability to resolve a signal from the background does not specifically address the ability to quantify at that level.) These instrument detection limit values ranged from 0.03 to 2.3 ng/mL, depending on the analyte. Later work, during phase C, was performed to determine the method detection limits, i.e., when the analyte compound is extracted from a real sample containing many other compounds.

b. Battelle-Columbus Division

A reported "limit of quantification" corresponded to the lowest concentration of standard used to prepare the calibration curve for an analyte. The values ranged from 4 to 60 ng/mL, depending on the analyte.

(1) The detection limits for the two chromatography-MS laboratories (AWL and BCD) were determined using different approaches, even though both were analyzing semivolatile organics. In this case, where there was interest in comparing two analytical methods, a common interlab definition and measure of detection and quantification limits was desirable. However, it is important for OB/OD that the laboratories determined their detection limits and made known their respective definitions. The difference in definitions did not cause any unsurmountable difficulty in comparing the test data from the two laboratories.

c. Lawrence-Berkeley Laboratory

LBL based the detection limits on three times the standard deviation observed from analyzing standards. These limits ranged from 2 to 150 ng/cm², depending on the element.

d. Oregon Graduate Center

The threshold of the GC-system integrators was the limiting factor in determining the lower detectable limit. Approximately 0.2 µg/m³ of a volatile hydrocarbon could be detected in air samples collected in 6-L canisters.

e. Sandia National Laboratories

SNL based the detection limits for gases on a signal-to-noise ratio of 2. As seen in Table 2.5c, the limits ranged from 2 ppbv to 2 ppmv, depending on the particular real-time instrument.

f. Sunset Laboratory

SSL determined detection limits for analyses of organic and elemental carbon by performing many instrument blank analyses (analyses performed on a filter punch aliquot that was known to contain no carbon). The standard deviation of the blank was approximately 0.25 μg of carbon per cm^2 of filter.

2.3.1.9.3 AWL Method Detection Limits for Soil Analysis

- a. Because of problems in extracting "dirty" soil samples and the presence of many interfering compounds in the analyses, the detection limits of analyte compounds in soil were appreciably higher than for the same analyte compounds in pure solvents.
- b. For comparison purposes and for SFC/MS analyses, Table 2.6 presents the detection limits for the semivolatile target analyte compounds in acetonitrile solvent and the method detection limits (or limits of quantification) for the same compounds in 400 grams of DPG test-site soils. Also shown are the corresponding ppb (by weight) of the analyte compounds in the 400-gram soil sample.

Table 2.5 Analyte List, Limits of Detection, and Limits of Quantification for phases A, B, and C Tests.

Chemical Analyte	Analyte List			Limit of Detection in Acetonitrile ng/mL*	Limit of Detection in 400g of DPG Test Site Soils. ^b		
	Phase A	Phase B	Phase C		ppbW	ng/mL	
2,4-Dinitrotoluene	TNT, M30	TNT, MR	TNT, Comp-B, RDX, Exp-D, M6, M1, MR	0.07	1	400	
2,6-Dinitrotoluene				0.05	1	400	
2,4,6-Trinitrotoluene				0.06	1	400	
2-Nitronaphthalene				0.03	1	400	
N-Nitrosodiphenylamine				0.12	10	4000	
1,3,5-Trinitrobenzene		TNT		0.12	10	4000	
2-Nitrodiphenylamine		MR		0.10	10	4000	
1-Nitropyrene		TNT		1.30	10	4000	
Naphthalene		TNT, MR		0.62	50	20000	
Benz[a]anthracene				0.73	1	400	
Benzo[a]pyrene				2.30	1	400	
Pyrene		MR		0.83	1	400	
Phenol ^c		TNT, MR		0.23	1000	400000	
Dibenzofuran				0.31	1	400	
Diphenylamine				0.21	1	400	
4-Nitrophenol				0.30			
Biphenyl				1.15			
Phenanthrene				1.20			
1-Methylnaphthalene				0.62			
2-Methylnaphthalene				0.62			
Ethyl centralite ^d	M30						
Nitroglycerin	MR	MR	0.21	10	4000		
Nitroguanidine ^e							
4-Nitrodiphenylamine	MR						
1,3,5-Trinitrophenol	Exp-D	0.35	20	8000			
RDX, Cyclonite ^f	Comp-B, RDX	0.20	1	400			
HMX, Octogen ^g							

*Detection limit for SFC/MS under chemical ionization/selected ion monitoring (signal/noise = 3).

^bBased on an acetonitrile extraction of 400 g of soil and then evaporation of the extract to a 1 mL sample for assay. Based on signal to noise ratio of 3 with respect to the soil background.

^cAnalyzed with GC/MS on phase A.

^dChemical name: N,N'-diethyl-N,N'-diphenylurea.

^eAnalysed as its methyl derivative.

^f1,3,5-Trinitrohexahydro-1,3,5-triazine.

^g1,3,5,7-Tetranitrooctahydro-1,3,5,7-tetrazocine.

2.3.1.9.4 Calibration Procedures

All of the instruments were fully calibrated prior to use, and calibration was checked at least daily to correlate instrument readings with analyte concentrations through a valid response function. Some procedures included additional calibration checks during a day's run.

a. Alpine West Laboratory

AWL ran a full calibration at the start of the project to verify linearity. A mass calibration was performed daily. A standard containing the analytes was run at the beginning of each day to establish the response factors. A two-component internal standard was introduced into each extracted sample. Project personnel ran blanks containing the internal standards every third day.

b. Battelle-Columbus Division

BCD ran a five-point calibration covering three orders of magnitude of concentration to establish linearity of response relative to the included internal standard. Calibration for quantification was performed before and after each group of samples (i.e., several times a day) using a standard containing single concentrations of each analyte. (The analytes that were included depended on whether negative or positive ion mode was to be employed for the samples.) They used a moving average response factor based on two analyses of the calibration standard. The MS was mass-calibrated daily. An instrument blank and/or method (extraction and concentration) blank containing only internal standard was analyzed at least once each day.

c. Lawrence-Berkeley Laboratory

LBL ran a standard filter containing single concentrations of S, Cu, and Ag daily. Absolute calibrations had been previously determined. The daily values were used to normalize sample results for any small day-to-day variation in output intensity of the x-ray tube.

d. Oregon Graduate Center

OGC ran a three-point calibration standard (plus blank) in duplicate and determined response factors. At the beginning of each day, three analyses were made of a single-point neohexane working standard. If the results fell within ± 2 percent, they used the average response to calibrate the analyses. If the three initial values exceed ± 2 percent, a fourth and fifth analyses were performed. A single-point calibration check standard was included after every fourth sample to check for calibration drift. Project personnel added an internal standard to every fourth sample.

e. Sandia National Laboratories

SNL calibrated real-time instruments before and after each test with NIST standard gases, and made daily checks of zero, flow, and span.

f. Sunset Laboratory

SSL performed a multipoint calibration approximately every 100 samples. Single-point calibration checks were done every 30 samples. An internal calibration standard was introduced into every sample. They ran instrument blanks every 30 samples.

2.3.1.10. Data Evaluation

2.3.1.10.1 Quality Control

A single OB/OD program coordinator was not appointed for monitoring QC for the chemical analysis procedures. The conduct of the laboratory phases of the BB study was the responsibility of the respective principal investigators. A variety of mechanisms was used to maintain quality control over the chemical analysis processes and/or systems. In addition to frequent calibration checks, as already noted, travel blanks (consisting of unexposed filters and resin cartridges), were shipped with each group of test-site samples. Their analysis-result values were used as sampling media background levels and to help interpret the results of analyzing actual trial samples. Each laboratory employed method blanks to help evaluate the background level of target analytes that

might be introduced with reagents used, by handling within the laboratory, or by other mechanisms. The values for method blanks were properly subtracted before reporting an observed concentration or quantity value for each actual trial sample. Each analytical laboratory LOI includes points related to the laboratory's internal QC program, as outlined below. Table 2.7 summarizes major features of each laboratory's internal QC procedures.

Table 2.6 Summary of Laboratory Calibration and Quality Control Procedures.

Laboratory*	Calibration Standards	Internal Standard	Control Spikes	Other Controls
AWL	Beginning of each day	Introduced into each extracted sample	Spiked samples run every two days	Instrument blanks; extraction efficiency
BCD	Beginning of each day	Introduced into each extracted sample	Every 2-6 samples; end of every day	Instrument blanks beginning of each day, every 2-6 samples; extraction efficiency
LBL	Run daily; contains S, Cu, and Ag	None	Run daily; contains 19 elements	Filter blank run every tray of samples
OGC	Every 4th sample	Every 4th sample	Daily	
SNL	Calibrated with a known conc. of O ₃ , NO _x , CO, CO ₂ , and SO ₂ before and after test	None	None	Daily check of zero, span, and flow rate
SSL	Introduced with each sample	Introduced with each sample	Known amount of carbon from sucrose standard solution	Single-point checks every 30 samples; instrument blanks every 30 samples

*AWL - Alpine West Laboratories; BCD - Battelle-Columbus Division; LBL - Lawrence-Berkeley Laboratory; OGC - Oregon Graduate Center; SNL - Sandia National Laboratories; SSL - Sunset Laboratory.

a. Alpine West Laboratory

Dr. Christine Rouse, Dr. Karin Markidis, and Dr. Milton Lee were responsible for SFC/MS QC measures. If response factors from the daily standards differed from historical values by more than 10 percent, they recalibrated. If an internal standard differed from the known value by more than 10 percent, the MS system automatically rejected the value. Spiked control standards were analyzed at least every other day. They consisted of 300 pg of 1-nitronaphthalene-d₇, and 240 pg of 9-phenylanthracene.

b. Battelle-Columbus Division

An institutional QA unit at BCD acted in an advisory role, and Dr. Laurence Slivon, project director, was in charge of QC for the analysis of BB samples. At the beginning of each analysis day and at intervals throughout the day, BCD analyzed a standard that contained all the target compounds and plus an internal standard, along with blanks that contained the internal standard.

c. Lawrence-Berkeley Laboratory

Dr. Robert Giauque was responsible for the QC program at LBL. In addition to the daily calibration check, LBL ran a control filter and a blank filter each day. The control filter contained known quantities of 19 elements. The results from the control filter were used to verify the stability of the x-ray spectrometer system and the consistency of the overall analysis. The ratio of the observed values to the standard values for each of the 19 elements were plotted. They used three-sigma control limits as the criteria for rerunning the day's samples. In the previous 2 years of operation, deviations beyond three sigma limits only occurred following a major malfunction of the system.

d. Oregon Graduate Center

Dr. Reinhold Rasmussen managed the QC responsibilities. A calibration check standard was run after every fourth sample. An internal standard was added to every fourth sample. Humidified zero air certification served as the blank for the calibration/check process.

e. Sandia National Laboratories

The QC officer for the OB/OD study was Mr. Wayne Einfeld. All instruments were either calibrated or underwent zero and span checks prior to each trial. Flow checks were made immediately prior to and following each trial.

f. Sunset Laboratory

Mr. Robert Cary performed all analyses as well as being the QC supervisor. He observed the automatic carbon spikes at the completion of each sample run. If the value did not fall within a certain specified range, it indicated that the unit was malfunctioning. If the single-point calibration standards (inserted every 30 samples) were not within ± 5 percent of the known value, sections of the analysis equipment were checked, adjusted, repaired, or replaced, as needed.

2.3.1.11. Preventive and Remedial Maintenance

Maintenance at the analytical laboratories throughout the OB/OD study resulted in minimal time being lost because of equipment malfunctions. BCD performed special cleanup and maintenance before commencing with the BB trial samples. AWL started with a newly-purchased SFC/MS. Some problems were experienced with the SFC/MS, but these were the result of the instruments' advanced design rather than to poor preventive-maintenance procedures.

2.3.2. Field Tests A, B, and C

Many concepts and procedures used during the BB test were applied during field testing and will not be reiterated. This section highlights the areas that were quite different and several activities that were changed. And, as mentioned earlier, all of the field studies (phases A, B, and C) were designed to be similar. New elements included preparation of the explosive materials in the thin-walled steel cylinders and propellants in heavy steel burn pans, performed by the DPG personnel was also new. The DPG personnel followed standard Army procedures in the placement and initiation of the explosive and propellant materials. Mr. Wayne Ursenbach, a private consultant,

provided expert advice on the placement and initiation of the test materials and developed LOI's for demolition and burn test activities.

2.3.2.1. Documentation and Data Management

2.3.2.1.1 The sample numbering scheme was refined to provide more meaningful (mnemonic) information. The scheme used for phase B is described in ELI document, Sample Numbering Key, Rev. 2. The mnemonic scheme used for phase C was more detailed and is included in LESC document, Soil and Fallout Sampling, 1 August 1990 (Field Test Series A, B, and C, Volume 2, Part B, Appendix B).

2.3.2.1.2 The system of handling data, previously described in Paragraph 2.3.1.5.1 was used for the BB test and phases A and B. Because the system was somewhat cumbersome and incurred some delay in the transmittal of technical information, it was revised for phase C. During phase C, the originators of all technical documents sent the original to ARC, which made official distribution of copies to all other interested parties. The originals were then sent to ELI for archiving. The complete official OB/OD file is now maintained by the PM.

2.3.2.2. Sampling and Real-Time Measurements

The procedures involved in handling and operating the sampling and measuring instruments on the aircraft were very similar to those applied during the BB Test. The primary sampling difference was the short collection period while the aircraft was in the plume. Major activities introduced during field testing were the sampling of soils, fallout, burn-pan residue, and sample handling, compositing, and weighing.

2.3.2.3. Analysis

During phases A and B, the weighing of filters was performed by AWL. To reduce the time required to process the filters, SNL developed a new LOI which specified that the filters should be weighed at DPG as soon as possible after the aircraft landed at Michael Army Airfield (the DPG

airfield). Laboratory analyses procedures remained essentially the same, except for preparing samples for extraction.

2.3.2.4. Letters of Instruction

The LOI's used in phases A, B, and C are listed in Volume 2, Part B, Appendix B. Revisions and additions made by SNL during the phase C dealt with the filter weighing procedure. Changes made by AWL during phase C reflect refinements in procedures for extracting and analyzing soil samples.

2.4. Quality Assurance - External Quality Control

2.4.1. Environmental Lab, Inc.

2.4.1.1. ELI, as the QA contractor supporting OB/OD, held a continuous and prime responsibility beginning with the BB test and continuing throughout phases A, B, and C. Dr. Gary Booth, chief executive officer of ELI, was the quality assurance director for the OB/OD project during these test segments. He was assisted by his staff (Table 2.1a) in executing a broad program of QA activities.

2.4.1.2. Quality Assurance Project Plans (QAPP).

QAPP were developed by ELI to support all phases of the project. The QAPP for phase C is included in this report as Volume 2, Part B, Appendix C.

2.4.1.3. Letters of Instruction.

ELI worked closely with, and issued guidance to, the operational organizations as they developed their respective LOI's. The LOI prepared by ELI, Procedures for Writing of Letters of Instruction, is included in Volume 2, Part B, Appendix B.

2.4.1.4. Sample Tracking System.

ELI also developed and instituted a sample tracking system, which was used very effectively in tracking and accounting for all of the samples taken during the project (except the gas sampling by the continuous, i.e., real-time, gas monitors used during the BB test and phases A, B, and C). The system was, in effect, a chain-of-custody system, enabling the tracking of each sample from the time it was taken through the final reporting of the results. Subsequent to the BB test, the system was refined to properly provide for the splitting and compositing of samples. The tracking system is described by the use of special forms.

2.4.1.5. Sample Numbering.

Beginning with phase B, an elaborate system of sample number designation (mostly mnemonic) was developed to handle the large number of samples in phases B and C and to be able to identify the sample designation from the origin of the sample. This system of sample numbering or designation is fully explained in Volume 2, Part B, Appendix B.

2.4.1.6. Informal Protocols

Although not fully documented as policy, most organizations involved in handling and analyzing samples generally understood the following:

2.4.1.6.1 No excess sample materials would be discarded after field sampling or in the laboratory.

2.4.1.6.2 No unused sample material or sample extractants would be discarded until authorized or directed by the OB/OD program manager.

2.4.1.6.3 All samples and sample extracts would be stored under refrigeration, except during actual extraction and analysis operations.

2.4.1.7. Coordination with the TSC.

As previously mentioned, Dr. Booth, as a member of the TSC, provided important coordination between the technical and quality assurance activities of the OB/OD project.

2.4.1.8. Systems audits.

Members of the ELI staff conducted periodic quality systems audits of the operational field and laboratory organizations. The general outline or format of the ELI systems audits covered the following topics:

2.4.1.8.1 LOI status.

2.4.1.8.2 Field/lab sampling

2.4.1.8.3 Field/lab analysis.

2.4.1.8.4 Instrument/method calibration.

2.4.1.8.5 Preventive/corrective maintenance.

2.4.1.8.6 Internal QC procedures.

2.4.1.8.7 Sample preparation and storage.

2.4.1.8.8 Preparation and use of spiked samples.

2.4.1.8.9 Instrument/equipment selection and use.

2.4.1.8.10 Determination of detection limits/limits of quantification.

2.4.1.8.11 Sample handling and transportation.

2.4.1.8.12 Data reduction and analysis.

2.4.1.8.13 Logbooks.

2.4.1.8.14 Personnel working with samples.

2.4.1.8.15 Building diagrams.

2.4.1.8.16 Research journals.

2.4.1.8.17 Chain-of-custody procedures.

2.4.1.8.18 Overall assessment/recommendations.

2.4.1.8.19 Additional comments.

The major results of these systems audits are discussed in Section 3.

2.4.2. Environmental Protection Agency (EPA)

2.4.2.1. Office of Solid Waste (OSW)

2.4.2.1.1 At the beginning of the OB/OD project, the program manager at AMCCOM, Rock Island Arsenal, Rock Island, Illinois requested support from EPA's Office of Solid Waste (OSW).

2.4.2.1.2 Mr. Chester Oszman, EPA Office of Solid Waste, has been a continual participant in the OB/OD program. During the preparation of the original test design plan, Mr. Oszman provided guidance as to the future direction of Resources Conservation and Recovery Act (RCRA) subpart X. Mr. Oszman continually provided guidance on how the OB/OD study could best meet EPA requirements for obtaining OB/OD permits. As results emerged from BB and field testing, Mr. Oszman arranged for a series of technology transfer seminars to be conducted within each EPA region and invited the PM to present the procedures and techniques used in collecting and assaying samples, and in analyzing laboratory data. These seminars offered the "forum for frank discussions and interchanges of ideas and opinions" and had a singularly beneficial effect on Federal and State regulators' acceptance of the OB/OD program's technology. Mr. Oszman continues to provide advice and offer assistance and support to the program.

2.4.2.2. Atmospheric Research and Exposure Assessment Laboratory of the EPA Office of Research and Development

2.4.2.2.1 The Research Monitoring and Evaluation Branch (RMEB) of EPA's AREAL at Research Triangle Park, NC, was uniquely qualified, capable, and experienced to provide the requested technical advice and quality assurance oversight, pertinent to the goals and activities of the OB/OD project.

2.4.2.2.2 The AREAL conducts intramural and extramural research programs through laboratory and field research with particular emphasis on atmospheric pollutants. The mission of the AREAL is to characterize, quantify, and control the exposure of humans, ecosystems, and materials to air pollutants. To fulfill their mission, AREAL conducts research and development and provides technical support in such areas as test methods for pollutants; emission inventories; QA reference materials, procedures, and guidelines; procedures for assessing quality of data; and dispersion models.

2.4.2.2.3 Quality Assurance Division (QAD).

Specific support to the OB/OD program has been provided by the RMEB personnel within the QAD. The QAD develops and conducts the EPA-wide air pollution quality assurance program through the following activities:

- a. Development of materials, systems, and procedures to assess the quality of air measurement data submitted to EPA,
- b. Standardization of methods for measuring pollutants in the ambient air and in stationary source emissions by evaluating, improving, and carefully describing methodology for these pollutants,
- c. Implementation of the EPA program for formal designation of reference and equivalent methods in support of the National Ambient Air Quality Standards and coordination of the development of the quality assurance requirements of EPA monitoring regulations,

- d. Evaluation of the quality of ambient air and stationary source measurements data reported to EPA,
- e. Preparation of guideline and technical assistance documents and provision of technical assistance on air pollution quality assurance matters to program and regional offices, states, and local air pollution agencies,
- f. Review of laboratory projects to determine appropriateness of quality assurance requirements.
- g. Conduct performance and systems audits on environmental monitoring projects.

2.4.2.2.4 RMEB Personnel.

The following EPA/AREAL personnel have been involved. Dr. William J. Mitchell, acting deputy director of AREAL, has been an active member of the OB/OD TSC throughout the entire study, providing technical advice on monitoring aspects of the program. Raymond C. (Rocky) Rhodes, quality assurance specialist, has been an active member of the working group, providing advice on quality assurance matters. Other members of the RMEB (Linda Porter, Elizabeth Hunike, Lisa Smith, Jack Bowen, Oscar Dowler, Howard Crist, Ellen Streib, Avis Hines, and William Barnard) participated in the conduct of quality systems audits (QSA's) and performance audits (QPA's) of OB/OD activities. Quality systems audits consist of a critical review of the written procedures and actual work activities of specific areas to provide a qualitative assessment of the capability to produce data of acceptable and known quality. Quality performance audits involve challenging the measurement systems with materials or devices of known characteristics to determine, quantitatively, the capabilities of the measurement systems to produce accurate and precise data.

2.4.2.2.5 RMEB Responsibilities.

Support for OB/OD by the RMEB has involved the review of test design plans, detailed test plans, QAPP's, LOI's (a procedural document used in lieu of the less flexible standard operating procedure), and test data, with appropriate comments and recommendations submitted to the OB/OD program manager. In addition, the RMEB has provided several activities of direct support

to the QA functions of OB/OD because of its experience and readily-available capability in conducting quality systems audits and quality performance audits. As an independent function and supplement to the activities of the QA director for OB/OD, RMEB conducted quality systems audits at the following facilities during field operations: Sandia National Laboratory, Dugway Proving Ground, Lockheed Engineering and Sciences Company, Alpine West Laboratory, Oregon Graduate Center, and Battelle-Columbus Division.

2.4.2.2.6 Other RMEB Support.

Also, various performance audits were conducted on the continuous gas analyzers (CO, CO₂, O₃, NO, NO₂, NO_x, and SO₂) and the flow measurement systems of the SNL aircraft equipment used for the BB and phases A, B, and C of the OB/OD program. Further, spiked samples of DPG soil, sampling canisters, sampling tanks, and sampling cartridges have been prepared by the RMEB for analyses at the various analytical laboratories. Results from these performance audits are presented in some detail in Section 3.2, providing independent assessments of the data quality from the laboratories.

2.4.2.2.7 AREAL Continuing Support.

Active and continuing support of these activities were provided by the AREAL laboratory throughout the OB/OD program. The accuracy, precision, and validity of the test data leads to reliable conclusions from the OB/OD program, a prime requisite for decisions to be made by the permit-granting authorities. Details of the results of the systems audits conducted by the RMEB of EPA are presented in Section 3.1 and the results of performance audits conducted by EPA are reported in Section 3.2.

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SECTION 3. QUALITY ASSURANCE ACTIVITIES

3.1. Systems Audits

3.1.1. General

Quality systems audits were conducted by ELI and by EPA throughout the testing, sampling, laboratory assay, and data analyses activities of the OB/OD study. The auditors examined compliance with documented procedures - primarily letters of instructions (LOI's) - which had been previously reviewed and approved by the program manager (PM) in consultation with the OB/OD scientific advisor, Technical Steering Committee (TSC), and ELI. When appropriate, they recommended changes they felt would enhance sample collection and subsequent analyses. The auditors also looked at overall laboratory practices including maintenance of logbooks, instrument calibration and preventive maintenance, and sample handling. When changes to existing operations were recommended, follow-up visits ensured that they were being implemented, or that sound rationale existed for continuing with the in-place system.

3.1.2. Schedule

The schedule of systems audits as conducted by EPA and ELI is given in Table 3.1.

Table 3.1a Schedule of Systems Audits.

Subject*	Test	ELI		EPA	
		Auditor	Date	Auditor	Date
BCD	BangBox	Parrish	12Dec88		
OGC	BangBox Phase B	McMullin McMullin	24Jan89 18Jan90	Christ	7Nov89
SSL	BangBox Phase A Phase B	McMullin McMullin McMullin	23Jan89 9May89 17Jan90		
LBL	BangBox	McMullin	31Jan89		
LESC	Phase C	Rhodes	6-19 Aug 90	Porter, Hunike, Hines Porter, Streib	6-27 Aug 90 20Feb90
ELI	Phase C			Porter, Hunike, Hines	6-27 Aug 90
SNL	BangBox	McMullin	16-17 Dec 88	Mitchell, Hunike, Porter, Rhodes	30 Nov - 2Dec88
	Phase B	McMullin Parrish	13-19 Oct 89	Mitchell	16-17 Oct 89
				Porter Rhodes	17-20 Oct 89 23-27 Oct 89
	Phase C			Smith, Hunike	16-26 Oct 89
				Barnard, Dowler	13-15 Oct 89
				Barnard, Dowler	6-10 Aug 89
				Barnard, Bowen Hines Hunike, Porter	27Aug90 6 Aug - 19Sep90

Table 3.1b Schedule of Systems Audits (Cont'd).

Subject*	Test	ELI		EPA	
		Auditor	Date	Auditor	Date
AWL	BangBox	Parrish	18Jan89		
	Phase A	McMullin	17Jul89		
	Phase B	Parrish	9Feb90	Hunike, Rhodes	19Oct89
				Hunike, Rhodes, Smith	24Oct89
				Porter, Smith	5-6 Dec 89
				Rhodes, Smith	20Feb90
	Phase C			Porter, Rhodes, Streib	21Feb90
				Hunike, Porter	16Aug90
				Mitchell, Porter	15Nov90

*AWL - Alpine West Laboratories; BCD - Battelle-Columbus Division; OGC - Oregon Graduate Center; SSL - Sunset Laboratory; LBL - Lawrence-Berkeley Laboratory; LESC - Lockheed Engineering and Sciences Company; ELI - Environmental Labs, Inc; SNL - Sandia National Laboratories.

3.1.3. Audit Administration

3.1.3.1. General

The auditors from both EPA and ELI were well qualified to assess scientific instruments and technical procedures. All were knowledgeable and experienced in good QA/QC practices, had scientific education and training backgrounds, and were familiar with the technical and scientific audits being conducted. Their use of preplanned questionnaires, check lists, and sequences provided a complete and impartial basis for assessing facilities and operations.

3.1.3.2. ELI-Conducted Audits

During each visit, ELI auditors followed the ELI outlined sequence described in Section 2 and used the checksheet illustrated in Volume 2, Part B, Appendix B. As individual audits progressed, the auditors shared their observations with project personnel to facilitate on-the-spot changes. Following their visits, the auditors prepared formal reports.

3.1.3.3. EPA-Conducted Audits

In conducting systems audits, the EPA auditors used preplanned questionnaires and check sheets. Because of their diversified experience in both developmental projects and on-going testing, the EPA auditors, in addition to their normal examinations, provided technical suggestions for modifying sampling and analytical procedures. At conclusion of each visit, the auditors briefed project personnel on their findings, and subsequently preparing their formal reports.

3.1.4. Evaluation

Throughout the OB/OD study, EPA and ELI reported results of their audits to the PM. Tables 3.2 through 3.23 summarize results of systems audits. The findings discussed below are included in Volume 2, Part B, Appendix D.

3.1.4.1. Sandia National Laboratories

3.1.4.1.1 ELI-Conducted Audits

a. BB Test

During the initial BB trials in December 1988, ELI audited the SNL activities at Albuquerque, New Mexico. This was the first time the sampling equipment and real-time analyzers were assembled as a cohesive package for OB/OD test purposes, and the auditor directed the majority of his attention to QC procedure evolution and application in this unique test environment. A summary of ELI audit findings is in Table 3.2.

Table 3.2 Results of ELI^a Systems Audit of SNL^b in December 1988.

Test	Finding	Comment
BangBox	LOI's ^c for real-time analyzers complete.	No action required. Post-test assay of bubbler samples will follow standard analysis procedures.
	SNL sample-numbering system used in lieu of ELI numbering system.	No action required. Small number of samples will preclude any confusion.
	Instrument calibration conducted as per manufacturer instructions.	No action required.
	Instrument preventive maintenance conducted as per manufacturer recommendations	No action required.
	Data reduction and analysis LOI incomplete.	SNL will complete LOI before data analyses begins.
	Logbooks and journals properly maintained.	No action required.

^aEnvironmental Labs Inc.

^bSandia National Laboratories.

^cLetter(s) of instruction.

b. Phase A

Phase A was designed as an operational readiness inspection and the PM determined that an audit conducted by ELI was not necessary.

c. Phase B

An ELI audit of SNL activities at DPG during phase B testing produced satisfactory findings. Emphasis of this audit was on instrument calibration, journals and logbooks, and sample handling procedures. Auditors found that correct procedures were being followed. Results are summarized in Table 3.3.

Table 3.3 Results of ELI^a Systems Audit of SNL^b in October 1989.

Test	Finding	Comment
Phase B	LOI's ^c all available. Some variation in procedures due to field test environment.	Deviation from standard procedures will be documented in journals.
	All instruments calibrated. Logbooks available.	No action required.
	Instrument preventive maintenance conducted as per manufacturer instructions.	No action required.

^aEnvironmental Labs Inc.

^bSandia National Laboratories.

^cLetter(s) of instruction.

d. Phase C

Systems auditing activities of SNL by ELI during phase C consisted primarily of observing filter weighing operations. The auditor concurred with EPA findings which are delineated in paragraph 3.1.4.1.2d.

3.1.4.1.2 EPA-Conducted Audits

a. BB Test

During its audit of SNL during BB-related activities, the EPA team concluded that requirements of the test plan were being followed. Results are summarized in Table 3.4.

Table 3.4 Results of EPA^a Systems Audit of SNL^b During BangBox Testing.

Test	Finding	Comment
BangBox	All equipment properly operating and detonation trials conformed to test plan.	No action required.
	Few spare parts on hand for continuous gas analyzers.	SNL felt historical reliability of analyzers precluded need for extensive spare-parts inventory.

^aU.S. Environmental Protection Agency.

^bSandia National Laboratories.

b. Phase A

The EPA audit team concluded that SNL systems were operating properly and that records and documentation were being adequately maintained. Additional findings are contained in Table 3.5.

Table 3.5 Results of EPA^a Systems Audit of SNL^b During Phase A Testing.

Test	Finding	Comment
Phase A	NO ₂ , SO ₂ , and O ₃ monitors were not installed at time of audit.	Monitors were installed before testing began.
	No means to determine if vacuum in canisters has been maintained prior to sampling, and to determine if sample has been collected.	Requirement for canister vacuum gages relayed to OGC ^c for future testing.
	No inventory of spare parts for samplers on airplane.	SNL felt historical reliability of analyzers precluded need for extensive spare-parts inventory.

^aU.S. Environmental Protection Agency.

^bSandia National Laboratories.

^cOregon Graduate Center. OGC provided the evacuated sampling canisters operated by SNL.

c. Phase B

Four months after completion of phase A, the EPA audit team returned to Utah to audit SNL activities during phase B. Although the thrust of the audit was on performance, some systems observations were made. These observations are contained in Table 3.6.

Table 3.6 Results of EPA^a Systems Audit of Instruments Aboard SNL^b Aircraft During phase B Testing.

Test	Finding	Comment
Phase B	Vacuum gages had not been installed on canisters.	Vacuum gages were in route from OGC ^c to test site for installation prior to testing.
	DAS, probes, temperature and dew point instruments calibrated within 12-month period.	None required.
	A spare parts inventory had not been developed.	SNL felt historical reliability of analyzers precluded need for extensive spare-parts inventory.

^aU.S. Environmental Protection Agency.

^bSandia National Laboratories.

^cOregon Graduate Center.

d. Phase C

Results of the EPA system audits of SNL operations during phase C are contained in an interim report. These audits included aircraft operation, measurement systems and the sample collection, and filter weighing operations.

(1) Findings of the aircraft systems audit are summarized in Table 3.7. Deficiencies associated with aircraft operation and measurement centered on isolated temporary instrument and equipment malfunctions.

Table 3.7 Results of EPA^a Systems Audit of SNL^b During Week of 6 August 1990.

Test	Finding	Comment
Phase C	All gas analyzers and sample collection systems were operational during testing.	Minor mechanical adjustments to O ₃ and NO _x analyzers were required once, and made.
	Vacuum gages had been installed on canisters prior to start of testing.	None. Indication of canister sampling capability and completion assured.
	Air intake valve malfunctioned twice during field testing.	Repairs made within 24 h.
	Some computer input lines registered noise levels above normal.	Source attributed to prevailing high ambient temperatures. Muffin fans installed.
	Air intake valve malfunctioned twice during field testing.	Repairs were made within 24 h.
	A spare parts inventory had not been developed.	SNL felt historical reliability of analyzers precluded need for extensive spare-parts inventory.
	20 percent loss of NO ₂ in Teflon™ bag after 6 min.	Studies required to determine stability of compounds when contained in Teflon™ bags.

^aU.S. Environmental Protection Agency.

^bSandia National Laboratories.

(2) All of the filter-weighing equipment used during phase C testing was provided by SNL and had been relocated from Albuquerque, New Mexico, to DPG specifically for the test. The room at DPG designated for weighing filter samples was a temporary facility within a two-minute drive from the aircraft's parking site at Michael Army Airfield, thus allowing weighing operations immediately after sample collection. Although the room was small, it accommodated sufficient equipment and personnel to permit gathering accurate weight data during testing. The weighing operations were conducted as originally planned, but some minor modifications were made on-site to compensate for unanticipated conditions. A summary of these modifications and of shortcomings of the filter weighing facility which were resolved by test personnel, are indicated in Table 3.8.

Table 3.8 Results of EPA^a Systems Audit of SNL^b Filter Weighing During Week of 6 August 1990.

Test	Finding	Comment
Phase C	Computer/balance interface software not functioning properly.	Computer was disconnected and weights recorded manually in logbook.
	Aluminum foil was substituted for LOI ^c -specified Teflon™ sheets to hold filter samples during conditioning.	None required. Teflon™ sheets caused static electricity which interfered with weighing process.
	Sample test series needed to provide documented basis for filter conditioning and weighing procedures.	SNL will develop test in which filters are conditioned to constant weight before and after sampling.
	Weighing room located adjacent to soil-handling room, thus potentially subject to soil dust contamination.	Access to weighing room was restricted to essential personnel, thus reducing the potential of dust entering as the door was opened.

^aU.S. Environmental Protection Agency.

^bSandia National Laboratories.

^cLetter of instruction.

(3) An QA consultant was on-site at DPG throughout phase C testing. In general concurred with system-audit conclusions reached by EPA auditors.

3.1.4.2. Alpine West Laboratories

3.1.4.2.1 ELI-Conducted Audits

a. BB Test

The initial audit by ELI was conducted while AWL was determining detection and quantification limits prior to its analyses of the BB samples. The auditor found AWL operations satisfactory overall, but felt control of samples could be improved. His findings are summarized in Table 3.9.

Table 3.9 Results of ELI^a Systems Audit of AWL^b on 18 January 1989.

Test	Finding	Comment
BangBox	LOI's ^c covering OB/OD-related activities were complete and available.	No action required.
	Preventive maintenance is conducted regularly by a trained technician and recorded in instrument logbooks.	No action required.
	Solvent used for cleaning and extraction had been changed from acetonitrile to dichloromethane.	AWL agreed to revise the LOI to reflect use of dichloromethane for extraction.
	Instrument calibrations were conducted daily and recorded on unsigned forms.	Future calibration records will be signed.
	Samples in the laboratory were stored in an unlocked freezer in the office of the laboratory director.	None required. The AWL director stated that access to freezer was sufficiently restricted to assure security for OB/OD samples.

^aEnvironmental Labs Inc.

^bAlpine West Laboratories.

^cLetter(s) of instruction.

b. Phase A

A second systems audit was conducted while AWL was actively analyzing samples from phase A. The findings were predominantly positive, but once again contained a concern over control of some samples. Results of this audit are summarized in Table 3.10.

Table 3.10 Results of ELI^a Systems Audit of AWL^b on 17 July 1989.

Test	Finding	Comment
Phase A	Revised LOI's ^c had been assigned new numbers.	Prior versions will be voided to prevent any possible confusion.
	Analyses being conducted as specified in current LOI's.	No action required.
	Instruments were being properly calibrated.	No action required.
	Logbooks and journals were properly maintained.	No action required.
	Preventive maintenance was conducted as specified by instrument manufacturers.	No action required.
	Some unprocessed samples were stored in a freezer not directly controlled by AWL.	AWL would try to accommodate samples in its freezer.

^aEnvironmental Labs Inc.

^bAlpine West Laboratories.

^cLetter(s) of instruction.

c. Phase B

The third systems audit of AWL by ELI followed the completion phase B testing. Ten LOI's governing AWL analytical support were on hand and all were current. The auditor, noting that AWL was no longer involved in sample collection, made the findings shown in Table 3.11.

Table 3.11 Results of ELI^a Systems Audit of AWL^b on 9 February 1990.

Test	Findings	Comment
Phase B	Filters were not being weighed three times as stipulated in LOI's ^c .	LOI procedures were followed during subsequent analyses.
	A complicated extract compositing procedure was used to prepare quasi-duplicate extracts for analysis.	LOI-specified procedure will be used for remaining duplicate sample analyses.
	The SFC-MS ^d was calibrated daily following procedures specified in two LOIs.	No action required.
	One chemist has been assigned responsibility of assuring internal QC procedures are followed.	No action required.
	All samples are now stored in freezers having restricted access.	No action required. This resolves sample control issues previously raised.
	Spiked samples are introduced daily for analysis.	No action required. This is an LOI-specified procedure.

^aEnvironmental Labs Inc.

^bAlpine West Laboratories.

^cLetter(s) of instruction.

^dSupercritical fluid chromatograph-mass spectrometer.

d. Phase C

There was no systems audit of AWL conducted by ELI during phase C operations.

3.1.4.2.2 EPA-Conducted Audits

a. BB Test

No system audit of AWL was conducted by EPA during BB test activities.

b. Phase A

Phase A was designed as a DPG operational readiness inspection and, as such, no system audit of AWL was necessary.

c. Phase B

The first EPA-conducted systems audit of AWL was to assess the conformance of AWL personnel to LOI's and, particularly, to determine how filter samples were being conditioned and processed. Auditors visited AWL on three separate occasions in 1989: The first time to conduct the audit, the second time to reassess filter conditioning, and the final time to reaudit filter handling, conditioning, and extraction. During the concluding visit, the auditors felt that some improvements in filter processing and documentation had been made, but additional effort was required. Other procedures were found satisfactory. Results of these audits are summarized in Tables 3.12 through 3.14.

Table 3.12 Results of EPA^a Systems Audit of AWL^b on 19 October 1989.

Test	Finding	Comment
Phase B	Particulate matter on quartz filters was becoming dislodged during handling (but was recovered each time).	New handling procedures were developed and the LOI ^c revised accordingly.
	Temperature and relative humidity in the weighing room should be recorded.	A recording thermo-humidigraph was installed in weighing room.
	Lowest gradation of thermometer in freezer was -10 °C; one reading down to -20 ° would be preferred.	A thermometer with a lowest gradation of -20 °C was installed in freezer holding OB/OD samples.

^aUS Environmental Protection Agency.

^bAlpine West Laboratories.

^cLetter of instruction.

Table 3.13 Results of EPA^a Systems Audit of AWL^b on 24 October 1989.

Test	Finding	Comment
Phase B	Entries in the logbook for recording filter weights concerning dates, analyst, relative humidity, and temperature were not clear.	AWL agreed to enhance documentation of filter weighing.
	A small fan would assure uniform temperature and humidity in the weighing room. (No deviation from uniformity was recorded by auditors.)	AWL will check uniformity of temperature and humidity and take any necessary corrective action.
	Filters could be placed in a "V" open position.	AWL will determine if any particulate matter is being lost due to present flat configuration and take any necessary corrective action.

^aU.S. Environmental Protection Agency.

^bAlpine West Laboratories.

Table 3.14 Results of EPA^a Systems Audit of AWL^b During 5-6 December 1989.

Test	Finding	Comment
Phase B	Extraction and analyses records were well maintained.	No action required.
	Documentation of filter weighing required additional improvement.	Data sheets, where possible, were upgraded.
	Temperature and relative humidity were stable for a six-week period.	No action required.
	An intercomparison study between two balances used for filter weighing should be undertaken.	Two balances were correlated and found both accurate to 0.1 mg
	SFC-MS ^c and extraction operations were well documented.	No action required.

^aU.S. Environmental Protection Agency.

^bAlpine West Laboratories.

^cSupercritical fluid chromatograph-mass spectrometer.

The auditors returned to AWL in February 1990 to conduct a fourth phase B-related audit. The auditor found AWL's procedure for preparing duplicate samples complex, but did not recommend any change. The auditor concluded that temperature and relative humidity records, and found that weighing room had been adequately maintained within $\pm 5^{\circ}\text{F}$ and the humidity within ± 5 percent.

3.1.4.3. Battelle Columbus Division

BCD supported the OB/OD study only during the BB Test and was not involved with any field testing.

3.1.4.3.1 ELI-Conducted Audits

Shortly after the BB test was initiated, ELI conducted a systems audit at BCD. The auditor found equipment and procedures satisfactory and the report indicated no significant adverse findings. Observations are summarized in Table 3.15.

Table 3.15 Results of ELI^a Systems Audit of BCD^b on 12 December 1988.

Test	Finding	Comment
BangBox	LOI's ^c are complete and being followed.	No action required.
	Accurate sample-tracking procedure being used.	No action required.
	Instruments are calibrated as per LOI and recorded.	No action required.
	Freezers containing samples are not locked.	No action required. Although freezer cannot be locked because of high laboratory activity level, access is limited to authorized personnel. Room with freezer is locked at night.
	Instrument preventive maintenance program is well conducted.	No action required.
	Logbooks well maintained.	No action required.

^aEnvironmental Labs Inc.

^bBattelle Columbus Division.

^cLetter(s) of instruction.

3.1.4.3.2 EPA-Conducted Audits

No EPA systems audits were conducted at BCD due to its historically satisfactory performance on other EPA projects.

3.1.4.4. Oregon Graduate Center

3.1.4.4.1 ELI-Conducted Audits

a. BB Test

A systems audit was conducted by ELI at OGC while BB testing was underway. Because this audit was undertaken at a time when OGC was in the process of procuring equipment for the OB/OD study and was not analyzing OB/OD samples, the auditor did not evaluate OGC adherence to OB/OD-specific procedures. Results of this audit are summarized in Table 3.16.

Table 3.16 Results of ELI^a Systems Audit of OGC^b on 24 January 1989.

Test	Finding	Comment
BangBox	LOI(s) ^c were not completed.	Standard test procedures were being used pending completion of LOI's.
	Many empty sample jars shipped to OGC by AWL ^d , were either broken or had loose lids upon arrival.	AWL changed shipping procedures to preclude future damage.

^aEnvironmental Labs, Inc.

^bOregon Graduate Center.

^cLetter(s) of instruction.

^dAlpine West Laboratories. AWL was responsible for providing sterile jars to OGC for shipment of tank and canister extracts.

b. Phase A

Because the phase A test had been designated as an operational readiness inspection, an ELI audit of OGC was neither necessary nor conducted.

c. Phase B

The second systems audit of OGC by ELI at OGC was conducted while analyses of phase B samples were underway. The auditor noted that OGC was using a standardized testing procedure (a slight modification of an EPA method, TO-14) in lieu of an OB/OD-specific LOI. The laboratory director expected that the LOI under development would closely parallel the EPA method being used, and that the quality of data would not change upon LOI completion. The auditor concluded that quality control practices were good. A summary of audit findings is presented in Table 3.17.

Table 3.17 Results of ELI^a Systems Audit of OGC^b on 18 January 1990.

Test	Finding	Comment
Phase B	LOI(s) ^c not finalized.	Standard testing procedures were being used pending development of LOI's.
	Logbooks were not maintained for each major equipment item.	OGC agreed to develop logbooks as time allowed. Until then, the existing system of recording calibration runs with records of concurrent analysis runs would suffice.

^aEnvironmental Labs, Inc.

^bOregon Graduate Center.

^cLetter(s) of instruction.

d. Phase C

ELI did not conduct a systems audit of OGC during phase C testing.

3.1.4.4.2 EPA-Conducted audit

a. BB Test

The EPA did not conduct a systems audit of OGC during the BB test.

b. Phase A

The phase A test was designated an operational readiness inspection and an EPA-conducted audit was not necessary.

c. Phase B

The EPA audited OGC while OGC was analyzing phase B samples. The auditor determined that the measurement system used by OGC fully met technical standards and requirements and that valid OB/OD data of documentable quality would be produced. A synopsis of findings is included in Table 3.18.

Table 3.18 Results of EPA^a Systems Audit of OGC^b in November 1989.

Test	Finding	Comment
Phase B	One-point calibration checks are performed daily on an OB/OD-dedicated GC/FID ^c .	No action required.
	Dehumidified air samples run daily to check system cleanliness.	No action required.
	After cleaning, 2 percent of canisters are checked for contamination.	No action required.
	All canisters evacuated and stored 3-5 days, after which vacuum monitored as check for canister leaks.	No action required.

^aU.S. Environmental Protection Agency.

^bOregon Graduate Center.

^cGas chromatography/flame ionization detector.

d. Phase C

No EPA systems audit of OGC was conducted during phase C test support.

3.1.4.5. Lawrence Berkeley Laboratory

3.1.4.5.1 ELI-Conducted Audit

One quality systems audit was conducted by ELI at LBL during analysis of samples from the BB Test. The auditor concluded that the laboratory was well organized and operated. Observation are included in the following table. The only observation requiring corrective action is noted in Table 3.19. No other audits of LBL were conducted by ELI.

Table 3.19 Results of ELI^a Systems Audit at LBL^b on 31 January 1989.

Test	Finding	Comment
BangBox	Logbook/journal entries made in pencil.	No action recommended. Duplicate entries made in computer-generated journal.
	LBL XRFA ^c QC Procedures being used as LOI ^d	No action recommended. Procedures document, used by LBL for 30 years, submitted to ELI as OB/OD QC document.
	LBL-created system used to track samples in lieu of OB/OD-directed system.	No action recommended. LBL system was accurate and complete.

^aEnvironmental Labs, Inc.

^bLawrence-Berkeley Laboratory.

^cX-ray fluorescence analysis.

^dLetter of instruction.

3.1.4.5.2 EPA-Conducted Audits

EPA did not audit LBL because of prior satisfactory experience with LBL. The calibration and QC procedures used by LBL are described in "X-Ray Fluorescence Analysis of Environmental Samples", an article edited by the late Dr. T.G. Dzubay who was an EPA authority on the x-ray fluorescence analysis.

3.1.4.6. Sunset Laboratory

3.1.4.6.1 ELI-Conducted audits

- BB Test

The audit conducted at SSL during analysis of samples from the BB Test produced positive findings. The auditor noted some deviations from OB/OD documentation, but did not believe they were serious flaws. Results of this audit are summarized in Table 3.20.

Table 3.20 Results of ELI^a Systems Audit of SSL^b in January 1989.

Test	Finding	Comment
BangBox	LOI ^c had not been prepared due to unfamiliarity with intended scope of an LOI.	Auditor explained LOI purpose and content. SSL made commitment to prepare LOI in immediate future.
	No logbook was maintained for automated computer analysis system.	Logbook not recommended. Computer prints calibration check with each sample analyzed.
	Sample logging procedures did not include all phases of sample handling.	SSL agreed to develop a sample-tracking form.

^aEnvironmental Labs Inc.

^bSunset Laboratory.

^cLetter of instruction.

b. Phase A

An audit of SSL phase A testing to ensure that SSL was prepared to accommodate any samples that might be submitted for analysis. At this time, SSL suggested baking the aluminum used in wrapping filters for shipment, thereby removing any residual carbon from the manufacturing process. The auditor concluded that SSL maintained high quality and accuracy standards. A synopsis of his report in Table 3.21.

Table 3.21 Results of ELI^a Systems Audit of SSL^b in May 1989.

Test	Finding	Comment
Phase A	LOI ^c had been prepared for review.	Auditor conveyed LOI to ELI Director for review process.
	The limited number of glass filters submitted for analysis (6) were melting during assay procedure.	Auditor made commitment to discuss issue with AWL. ^d (Glass filters were replaced with quarts in Phase B.)

^aEnvironmental Labs Inc.

^bSunset Laboratory.

^cLetter of instruction.

^dAlpine West Laboratories.

c. Phase B

The final ELI audit of SSL came shortly after phase B samples had been analyzed. The approved LOI was available and being used.

d. Phase C

No phase C samples were submitted to SSL for analysis.

3.1.4.6.2 EPA-Conducted Audits

EPA did not audit SSL due to historically satisfactory experience with the laboratory.

3.1.4.7. Lockheed Engineering and Sciences Company

3.1.4.7.1 ELI-Conducted Audits

a. BB Test

Lockheed did not participate in BB Test activities.

b. Phase A

In that phase A was an operational readiness inspection, LESC was not audited during phase A activities.

c. Phase B

No audit of LESC phase B activities was conducted by ELI.

d. Phase C

The first ELI audit of LESC activities was conducted during phase C testing. The auditor found that LESC operators were well-trained, followed LESC LOI's, and exercised good practices (Table 3.22).

e. EPA-Conducted Audits

The first EPA-conducted systems audit of LESC activities occurred during phase C testing. The auditors were quite laudatory of LESC employees and their performance. The only deficiency noted involved a technical consideration in weighing small soil (fallout) samples: In some cases the net weights of the fallout were negative. This weight loss was believed to have been caused by loss of weight of the sample jar/plastic lid in the extreme heat experienced at the test site. No firm conclusion was drawn, and this anomaly warrants investigation prior to initiation of the next OB/OD field test. Audit findings are summarized in Table 3.23.

Table 3.22 Results of ELI^a Systems Audit of LESC^b During phase C Testing.

Test	Finding	Comment
Phase C	LESC personnel closely followed prescribed practices.	No action required.
	Supervisory personnel were all very familiar with project support responsibilities.	No action required. Departure of a supervisor from test site did not incur any lost project time.
	Soil-handling room should be cleaned more frequently to reduce possibility of contaminating weigh room.	Soil-handling room cleaning schedule was accelerated.

^aEnvironmental Labs Inc.

^bLockheed Engineering and Sciences Company.

Table 3.23 Results of EPA^a Systems Audit of LESC^b Activities During phase C. Testing.

Test	Finding	Comment
Phase C	Field staff conscientious and knowledgeable.	No action required.
	Use of students could precipitate high turn-over and loss of skills.	Obviated by training program and "buddy" system pairing new with experienced workers.
	Occasional sample-bottle weight loss.	Samples removed from field bottles evidencing negative weight and weighed separately.
	Cleaning soil compositing equipment generated considerable dust in soil compositing room.	All sample bottles were covered during cleaning process.

^aU.S. Environmental Protection Agency.

^bLockheed Engineering and Sciences Company.

3.2. Performance Audits

3.2.1. General

Under normal circumstances, the organization responsible for project QA (ELI in this instance) initiates and conducts performance audits. However, ELI delegated responsibility for performance audits to EPA because of EPA's experience and capabilities in conducting this type audit and EPA's interest in, and need for, test results.

3.2.2. Schedule

The schedule in Table 24 outlines performance audits conducted by EPA during the BB Test and subsequent field testing.

Table 3.24 Schedule of Performance Audits Conducted by EPA^a.

Audited Organization	Test	Measurement Parameter Analytes Involved	Audit Dates	Report Cite ^b
Sandia National Laboratories	BangBox	Flow rates: Particulate and XAD-2™ resin samplers. Gas analyzers: CO, CO ₂ , O ₃ , NO/NO _x , SO ₂	Feb 89	1 Nov 89 31 Jul 89
	Phase A	Gas analyzers: CO, CO ₂	Jun 89	1 Nov 89
	Phase B	Flow rate: Aircraft probe Gas analyzers: CO, CO ₂ , O ₃ , SO ₂ , NO/NO _x	Oct 89	26 Jan 90
	Phase C	Flow rates Gas analyzers: CO, CO ₂ , SO ₂ , NO/NO _x	Aug 90	7 Sep 90
Alpine West Laboratories	BangBox	Spiked soil Spiked XAD-2™ cartridges Spiked 32-L tank extract Spiking solution	Dec 88	2 Mar 89 1 Nov 89
	Phase B	Spiked soil	Nov 89	26 Jan 90
	Phase C	Spiked soil		7 June 91
Battelle Columbus Division	BangBox	Spiked soil Spiked XAD-2™ cartridges Spiked 32-L tank extract	Dec 88	2 Mar 89 1 Nov 89
Oregon Graduate Center	BangBox	Spiked 6-L canisters Spiked 32-L tanks	Dec 88	2 Mar 89 1 Nov 89
	Phase B	Spiked 6-L canisters	Nov 89	26 Jan 90 13 Feb 90
	Phase C	Spiked 6-L canisters		7 June 91

^aU.S. Environmental Protection Agency

^bCitations are report dates. A more complete citation is included in report text.

3.2.3. Execution

An EPA audit team applied gases of known values to real-time gas analyzers and verified that instrument performance fully met EPA standards. The AREAL spiked atmosphere and soil samples, ultimately comparing assay results from the audited laboratories to the known values of these spikings. The audits encompassed all aspects of the OB/OD program, were thorough and detailed, and substantiated that procedures and instruments conformed to EPA standards. Results of these audits are contained in Tables 3.25 through 3.30.

3.2.3.1. Sandia National Laboratories

A variety of measurement systems were used by SNL during the OB/OD study. The measurements that were audited included flow rates for particulate and cartridge samplers, and the instrument responses for gas (CO, CO₂, O₃, SO₂, NO/NO₂) analyzers. Tables 3.25 and 3.26 summarize results of these audits. The O₃, NO, and NO_x analyzers were not audited during phase A because they were not fully installed in the aircraft when the audit was conducted. The NO analyzer was not audited during phase B because it was calibrated for SNL by the EPA audit team due to an inoperable SNL calibration system. The SO₂ analyzer was not used during phases B and C. With the exception of the CO₂ monitor in phase A, auditors found that all instruments were operating within QAPP target criteria (± 10 percent of the true value). The EPA considers results falling within ± 10 percent of known values to be within acceptable limits. In the judgment of the EPA auditors, the 14 percent positive bias in the CO₂ monitor in phase A did not impact on the data quality because the CO₂ formed in the detonation was determined as the difference between the ambient air and plume concentrations.

Table 3.25 Results of EPA^a-Conducted Flow Rate Audits of SNL^b Samplers.

Test	Instrument/Sampler Audited	Audit Results ^{c,d}
BangBox	semi-VOST ^e /Particulate sampler	Within $\pm 10\%$
Phase B	Particulate sampler (w/constrictor plate installed)	-2.9 %
Phase C	Particulate sampler (w/o constrictor plate or filter)	-2.7 %
	Airplane probe	Within $\pm 10\%$

^aU.S. Environmental Protection Agency.

^bSandia National Laboratories.

^cFlow rates, as indicated by SNL instruments, were compared to flow rates as measured by auditor-provided instruments.

^dAudit values reported as ± 10 percent fell within the ± 10 percent tolerances.

^eSemi-volatile organic sampling train.

Table 3.26 Results of EPA^a Audits of SNL^b Gas Analyzers (Deviation from Known Value).

Test	SO ₂ (%)	CO (%)	CO ₂ (%)	O ₃ (%)	NO (%)	NO _x (%)
BangBox	$\pm 10.0^b$	± 10.0	± 10.0	± 10.0	± 10.0	± 10.0
Phase A	NA ^c	+10.0	+14.3	NA	NA	NA
Phase B	NA	+6.0	-6.2	+1.7	NA	+0.1
Phase C	NA	± 10.0	± 10.0	± 10.0	± 10.0	± 10.0

^aSandia National Laboratories.

^bAudit values reported as ± 10 percent fell within the ± 10 percent tolerances.

^cNot audited.

3.2.3.2. Alpine West Laboratories

3.2.3.2.1 Media.

During the BB test AWL extracted and analyzed soil and XAD-2™ samples spiked by EPA. They also analyzed the extracts from 32L tanks that had been spiked by EPA and subsequently extracted by Oregon Graduate Center. For phase A, no spiked soil samples were used because the objective was to check out the sample collection procedures developed from the BB tests. Spiked soil samples were used in phases B and C. Ancillary tests were also done to evaluate the spiking and analytical methods being used in the OB/OD project.

3.2.3.2.2 Performance Audits.

a. BangBox

In December 1988, AREAL spiked 2 resins, 32-L tanks, and DPG soil samples. These, plus a spiking solution, were the basis of the first performance audit at AWL. All spikings were made in microgram quantities (5 to 36 µg) using chemicals from EPA's Quality Assurance Materials Bank (Reference 4). The 32-L tank was extracted by OGC and the extracts provided to AWL for analysis. Quantitative recoveries by both GC/MS and SFC/MS were achieved for the seven target analytes spiked on the soil samples, but very low recoveries were obtained from the resin's and the 32L tanks. As a result, the tanks and resins were dropped from the program. The seven analytes used were: phenol, 4-Nitrophenol, 2,4-Dinitrophenol, N-Nitrosodiphenylamine, benz(a)anthracene, benz(a)pyrene, and dibenz(a,h)anthracene. The soil samples were analyzed by AWL in August 1989 with the results shown in Table 3.27. These results obtained after 8 months of storage at -20° C provided evidence of the stability of the semivolatiles on soil samples in storage.

Table 3.27 AWL^a Analyses of Spiked Materials From the BangBox Test Series (Reported as Percent Deviation from Spiking Level).

Spiking Solution	Spiking Solution ^b	Spiked 32-L Tank ^{b,c}	Spiked Soil ^d	Spiked Soil ^e
Phenol	NR ^f	NR	-2.8	-2.7
4-Nitrophenol	-93	-93	1.8	0.0
2,4-Dinitrophenol	NR	NR	0.0	0.0
N-Nitrosodiphenylamine	-91	-80	1.8	0.0
Benz[a]anthracene	-26	4.3	0.0	0.0
Benzo[a]pyrene	-52	-41	2.9	5.9
Dibenzo[a,h]anthracene	NR	-60	1.9	4.0

^aAlpine West Laboratories.

^bSpiking level: 19-29 µg.

^c32-L tank extracted by Oregon Graduate Center; extract provided AWL.

^dSpiking level: 4.5-7.0 µg.

^eSpiking level: 23-36 µg.

^fNot reported.

NOTES:

1. Above results were obtained using supercritical fluid chromatography-mass spectrometry. Similar results were obtained with gas chromatography-mass spectrometry.
2. Assay of spiked resins is not reported. Pretreatment heating of resin material caused chemical degradation.
3. The spiking was done in December 1988. The soil extractions and assays were done in August 1989.

b. Phases B and C

Soil samples from Dugway Proving Ground were spiked with target analytes for phases B and C and these were subsequently analyzed as unknowns by AWL when they were processing the field samples. The phase B EPA-spiked soil studies involved microgram quantities (i.e., considerably more than were being encountered with test samples collected in the field). An additional EPA-spiked soil study was conducted for phase C at nanogram quantities, more closely approximating some of the levels being reported for test soil and filter samples, but at levels somewhat above the stated detection levels for the compounds. The spiking was performed by EPA, Research Triangle Park, NC in July 1990, and the results of the analyses were reported by AWL in March 1991. The analytes spiked on the soil samples in phases B and C were somewhat different from those used in the BangBox spiked soil samples, i.e., 1-nitropyrene, dibenzofuran, pyrene, 1,3,5-trinitrobenzene, 2,6-dinitrotoluene, 2,4-dinitrotoluene and naphthalene were added and N-nitrosodiphenylamine and the two nitrophenols were deleted from the original list. The spiking levels and the results are shown in Tables 3.28 and 3.29.

Table 3.28a Results for Samples Spiked by Slurry/Rotary-Evaporation Method, and Analyzed by SFC/MS Method^a.

Analyte	Sample #	Spiked(ng/sample)					Reported(ng/sample)			[(A-O)/O]x100 ^b (%)				
		5139	5137	5115	5109	5113	5139	5137	5115	5109	5139	5137	5115	5109
1-Nitropyrene	10100	4040	8080	40400	4040	14000	5100	7400	23000	4600	38.6	26.2	-8.42	-43.1
Dibenzofuran	428	1070	856	2140	1070	440	1100	940	1900	770	2.80	2.80	9.81	-11.2
Pyrene	384	7680	1920	19200	7680	560	6200	1800	14000	4500	45.8	-19.3	-6.25	-27.1
Diphenylamine	448	896	224	2240	1120	370	650	250	1400	430	-17.4	-27.5	11.6	-37.5
1,3,5-Trinitrobenzene	4000	2000	0	0	4000	2400	1300	230	250	2100	-40.0	-35.0	NA ^c	NA
2,6-Dinitrotoluene	400	200	5000	400	10000	250	130	3600	250	7600	-37.5	-35.0	-28.0	-37.5
Benz[al]pyrene	4000	8000	2000	4000	4000	3500	7100	2700	3500	930	-12.5	-11.3	35.0	-12.5
Naphthalene	40000	4000	40000	40000	4000	6700	1300	11000	9600	2200	-83.3	67.5	-72.5	-76.0
Phenol	40000	80000	80000	150000	120000	NR ^d	NR	NR	NR	NR	NR	NR	NR	NR
2,4-Dinitrotoluene	2000	5000	0	400	10000	1500	2600	340	670	6500	-25.0	-48.0	NA	-67.5
1,2-Benz[a]anthracene	980	392	784	3920	392	890	700	1100	3400	370	-9.18	78.6	40.3	-13.3

^aSamples (Dichloromethane solution) analysed SFC/MS with Finnigan MAT INCOS 50 MS.

^b(Assayed(A)-Spiked(O))/O)x100 = Percent deviation from spiking level.

^cNA denotes not applicable.

^dNR denotes not reported.

Table 3.28b Results for Samples Spiked by Slurry/Rotary-Evaporation Method, and Analyzed by GC/MS Method^a.

Analyte	Sample #	Spiked(ng/sample)					Reported(ng/sample)					$\frac{[(A-O)/O] \times 100}{}$ (%)			
		5139	5137	5115	5109	5113	5139	5137	5115	5109	5113	5139	5137	5115	5109
1-Nitropyrene	10100	4040	8080	40400	4040	11000	4100	9300	15000	3500	8.91	1.49	15.1	-62.9	-13.4
Dibenzofuran	428	1070	856	2140	1070	340	790	740	1600	790	-20.6	-26.2	-13.6	-25.2	-26.2
Pyrene	384	7680	1920	19200	7680	400	6700	1800	16000	6000	4.17	-12.8	-6.25	-16.7	-21.9
Diphenylamine	448	896	224	2240	1120	210	400	140	870	480	-53.1	-55.4	-37.5	-61.2	-57.1
1,3,5-Trinitrobenzene	4000	2000	0	0	4000	940	520	18	BD*	1000	-76.5	-74.0	NA ^c	NA	-75.0
2,6-Dinitrotoluene	400	200	5000	400	10000	270	190	3700	190	6400	-32.5	-5.00	-26.0	-52.5	-36.0
Benz[a]pyrene	4000	8000	2000	4000	4200	3000	7400	1300	3200	63	-25.0	-7.50	-35.0	-20.0	-98.4
Naphthalene	40000	4000	40000	40000	40000	7300	1600	14000	11000	1600	-81.8	-60.0	-65.0	-72.5	-60.0
Phenol	40000	80000	80000	150000	120000	NR*	NR	NR	NR	NR	NR	NR	NR	NR	NR
2,4-Dinitrotoluene	2000	5000	0	400	10000	1700	3400	360	850	6600	-15.0	-32.0	NA	113	-34.0
1,2-Benz[a]anthracene	980	392	784	3920	392	980	710	980	3700	550	0	81.1	25.0	-5.61	40.3

^aSamples (Dichloromethane solution) analysed GC/MS with Finnigan MAT INCOS 50 MS.

* $\frac{[(Assayed(A)-Spiked(O))/O] \times 100}{}$ = Percent deviation from spiking level.

^cNA denotes not applicable.

^dNR denotes not reported.

Table 3.28c Results for Samples Spiked by Slurry/Rotary-Evaporation Method, and Analyzed by GC/MS Method.

Analyte	#	Spiked(ng/sample)					Reported(ng/sample)					[(A-O)/O]x100* (%)					
		Sample	5139	5137	5115	5109	5113	5139	5137	5115	5109	5113	5139	5137	5115	5109	5113
1-Nitropyrene	10100	4040	8080	40400	4040	16000	3400	5400	18000	3700	58.4	-15.8	-33.2	-55.4	-8.42		
Dibenzofuran	428	1070	856	2140	1070	210	370	360	1300	490	-50.9	-65.4	-57.9	-39.3	-54.2		
Pyrene	384	7680	1920	19200	7680	410	5200	1400	16000	4500	6.77	-32.3	-27.1	-16.7	-41.4		
Diphenylamine	448	896	224	2240	1120	160	280	46	970	320	-64.3	-68.8	-79.5	-56.7	-71.4		
1,3,5-Trinitrobenzene	4000	2000	0	0	4000	2200	510	330	59	950	-45.0	-74.5	NA ^c	NA	-76.3		
2,6-Dinitrotoluene	400	200	5000	400	10000	240	160	3400	130	7700	-40.0	-20.0	-32.0	-67.5	-23.0		
Benzo[a]pyrene	4000	8000	2000	4000	3500	7800	1600	3200	BD ^d	-12.5	-2.50	-20.0	-20.0	-20.0	NA		
Naphthalene	40000	4000	40000	40000	4300	880	8500	10000	790	-89.3	-78.0	-78.8	-75.0	-80.3			
Phenol	40000	80000	90000	150000	120000	NR*	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	
2,4-Dinitrotoluene	2000	5000	0	400	10000	1800	3300	320	610	6600	-10.0	-34.0	NA	52.5	-34.0		
1,2-Benz[a]anthracene	980	392	784	3920	392	870	610	800	3400	630	-11.2	55.6	2.04	-13.3	60.7		

*Samples (Dichloromethane solution) analysed GC/MS with Finnigan MAT 8430 double focusing MS.

*[(Assayed(A)-Spiked(O))/O]x100 = Percent deviation from spiking level.

^aNA denotes not applicable.

^bBelow detection.

^cNR denotes not reported.

Table 3.28d Results for Samples Spiked by Slurry/Rotary-Evaporation Method, and Analyzed by GC/MS Method.

Analyte	Spiked(ng/sample)					Reported(ng/sample)					$[(A-O)/O] \times 100^b$ (%)				
	Sample #	5139	5137	5115	5109	5113	5139	5137	5115	5109	5113	5139	5137	5115	5109
1-Nitropyrene	10100	4040	8080	40400	4040	NA ^c	6400	NA	NA	6000	NA	58.4	NA	NA	48.5
Dibenzofuran	428	1070	856	2140	1070	NA	430	NA	NA	420	NA	-59.8	NA	NA	-60.7
Pyrene	384	7680	1920	19200	7680	NA	5200	NA	NA	4200	NA	-32.3	NA	NA	-45.3
Diphenylamine	448	896	224	2240	1120	NA	240	NA	NA	250	NA	-73.2	NA	NA	-77.7
1,3,5-Trinitrobenzene	4000	2000	0	0	4000	NA	1100	NA	NA	1700	NA	-45.0	NA	NA	-57.5
2,6-Dinitrotoluene	400	200	5000	400	10000	NA	120	NA	NA	6500	NA	-40.0	NA	NA	-35.0
Benz[a]pyrene	4000	8000	2000	4000	4000	NA	9400	NA	NA	BD ^d	NA	17.5	NA	NA	NA
Naphthalene	40000	4000	40000	40000	4000	NA	1200	NA	NA	1400	NA	-70.0	NA	NA	-65.0
Phenol	40000	80000	80000	150000	120000	NR ^e	NR	NR	NR	NR	NR	NR	NR	NR	NR
2,4-Dinitrotoluene	2000	5000	0	400	10000	NA	4000	NA	NA	8000	NA	-20.0	NA	NA	-20.0
1,2-Benz[a]anthracene	980	392	784	3920	392	NA	570	NA	NA	430	NA	45.4	NA	NA	9.69

*Samples (acetonitrile solution) analysed GC/MS with Finnigan MAT 8430 double focusing MS.

^b $[(\text{Assayed}(A)-\text{Spiked}(O))/O] \times 100$ =Percent deviation from spiking level.

^cNA denotes not applicable or sample not assayed.

^dBelow detection.

^eNR denotes not reported.

Table 3.28e Results for Samples Spiked by Aluminum Roll Method, and Analyzed by SFC/MS Method^a.

Analyte	Sample	Spiked(ng/sample)				Reported(ng/sample)				[(A-O)/O]x100 ^b (%)					
		\$110	\$107	\$134	\$129	\$116	\$110	\$107	\$134	\$129	\$116	\$110	\$107		
1-Nitropyrene	4040	12120	40400	4040	8080	5600	18000	54000	4000	7500	38.6	48.5	33.7	-0.99	-7.18
Dibenzofuran	1070	1070	428	3210	856	1000	1500	610	2500	990	-6.54	40.2	42.5	-22.1	15.7
Pyrene	7680	19200	1920	384	7680	5800	17000	1600	380	5300	-24.5	-11.5	-16.7	-1.04	-31.0
Diphenylamine	896	2688	448	896	224	360	80	200	690	220	-59.8	-97.0	-55.4	-23.0	-1.79
1,3,5-Trinitrobenzene	2000	0	4000	20000	4000	1500	160	1700	14000	21000	-25.0	NA ^c	-57.5	-30.0	-47.5
2,6-Dinitrotoluene	400	10000	400	200	5000	290	6700	220	110	3600	-27.5	-33.0	-45.0	-45.0	-28.0
Benz[al]pyrene	8000	5000	5000	2000	8000	5700	4400	3700	2200	5600	-28.8	-12.0	-26.0	10.0	-30.0
Naphthalene	40000	40000	4000	40000	40000	8500	13000	730	1700	8600	-78.8	-67.5	-81.8	-57.5	-78.5
Phenol	50000	40000	80000	230000	80000	NR ^d	NR	NR	NR	NR	NR	NR	NR	NR	NR
2,4-Dinitrotoluene	400	2000	10000	5000	0	890	1600	6200	3300	320	123	-20.0	-38.0	-34.0	NA
1,2-Benz[a]anthracene	392	980	3920	392	784	520	1200	2800	660	800	32.7	22.4	-28.6	68.4	2.04

^aSamples (Dichloromethane solution) analysed SFC/MS with Finnigan MAT INCOS 50 MS.

^b[(Assayed(A)-Spiked(O))/O]x100 = Percent deviation from spiking level.

^cNA denotes not applicable.

^dNR denotes not reported.

Table 3.2M Results for Samples Spiked by Aluminum Roll Method, and Analyzed by GC/MS Method.

Analyte	Sample #	Spiked(ng/sample)				Reported(ng/sample)				[(A-O)/O]x100 ^b (%)					
		\$110	\$107	\$134	\$129	\$116	\$110	\$107	\$134	\$129	\$116	\$110	\$107	\$134	\$129
1-Nitropyrene	4040	12120	40400	4040	8080	48000	12000	48000	40000	8600	18.8	-0.99	18.8	-0.99	6.44
Dibenzofuran	1070	1070	428	3210	856	850	950	380	2400	540	-20.6	-11.2	-11.2	-25.2	-36.9
Pyrene	7680	19200	1920	384	7680	7400	19000	1900	520	5600	-3.65	-1.04	-1.04	35.4	-27.1
Diphenylamine	896	2688	448	896	224	330	180	200	410	160	-63.2	-93.3	-55.4	-54.2	-28.6
1,3,5-Trinitrobenzene	2000	0	4000	20000	4000	680	35	1400	10000	1400	-66.0	NA ^c	-65.0	-50.0	-65.0
2,6-Dinitrotoluene	400	10000	400	200	5000	240	7300	180	110	3800	-40.0	-27.0	-55.0	-45.0	-24.0
Benzol[al]pyrene	8000	5000	5000	2000	8000	7500	4400	4200	1700	6500	-6.25	-12.0	-16.0	-15.0	-18.8
Naphthalene	40000	40000	4000	40000	13000	15000	1100	2000	9300	67.5	-62.5	-72.5	-72.5	-50.0	-76.8
Phenol	50000	40000	80000	230000	80000	NR ^d	NR	NR	NR	NR	NR	NR	NR	NR	NR
2,4-Dinitrotoluene	400	2000	10000	5000	0	1100	2100	7100	3500	380	175	5.00	-29.0	-30.0	NA
1,2-Benz[a]anthracene	392	980	3920	392	784	820	1200	4200	660	1300	109	22.4	7.14	68.4	65.8

^aSamples (Dichloromethane solution) analysed GC/MS with Finnigan MAT INCOS 50 MS.

^b[(Assayed(A)-Spiked(O))/O]x100 = Percent deviation from spiking level.

^cNA denotes not applicable.

^dNR denotes not reported.

Table 3.28g Results for Samples Spiked by Aluminum Roll Method, and Analyzed by GC/MS Method^a.

Analyte	Sample #	Spiked($\mu\text{g}/\text{sample}$)					Reported(ng/sample)			$[(A-O)/O] \times 100^b$ (%)					
		5110	5107	5134	5129	5116	5110	5107	5134	5129	5116	5110	5107	5134	5129
1-Nitropyrene	4040	12120	40400	40440	8080	1600	12000	54000	3700	7300	-60.4	-0.99	33.7	-8.42	-9.65
Dibenzofuran	1070	1070	428	3210	856	720	600	280	2300	440	-32.7	-43.9	-34.6	-28.3	-48.6
Pyrene	7680	19200	1920	384	7680	7000	18000	1600	450	5600	-8.85	-6.25	-16.7	17.2	-27.1
Diphenylamine	896	2688	448	896	224	340	180	160	290	150	-62.1	-93.3	-64.3	-57.6	-33.0
1,3,5-Trinitrobenzene	2000	0	4000	20000	4000	520	160	1300	15000	1300	-74.0	NA ^c	-67.5	-25.0	-67.5
2,6-Dinitrotoluene	400	10000	400	200	5000	150	7400	110	79	3800	-62.5	-26.0	-72.5	-60.5	-24.0
Benzof[<i>o</i>]pyrene	8000	5000	5000	2000	8000	8500	4000	5500	1800	7000	6.25	-20.0	10.0	-10.0	-12.5
Naphthalene	40000	40000	4000	40000	110000	12000	850	1800	8300	-72.5	-70.0	-78.8	-55.0	-79.3	
Phenol	50000	40000	80000	230000	80000	NR ^d	NR	NR	NR	NR	NR	NR	NR	NR	NR
2,4-Dinitrotoluene	400	2000	10000	5000	0	'900	1700	8500	3400	290	150	-15.0	-15.0	-32.0	NA
1,2-Benz[a]anthracene	392	980	3920	392	784	760	1400	3700	590	900	93.9	42.9	-5.61	50.5	14.8

^aSamples (Dichloromethane solution) analysed GC/MS with Finnigan MAT 8430 double focusing MS.

^b $[(\text{Assayed}(A)-\text{Spiked}(O))/O] \times 100$ = Percent deviation from spiking level.

^cNA denotes not applicable.

^dNR denotes not reported.

Table 3.28h Results for Samples Spiked by Aluminum Roll Method, and Analyzed by GC/MS Method^a.

Analyte	Sample #	Spiked(ng/sample)					Reported(ng/sample)					$\frac{[(A-O)-O]}{O} \times 100^b$ (%)				
		5110	5107	5134	5129	5116	5110	5107	5134	5129	5116	5110	5107	5134	5129	5116
1-Nitropyrene	4040	12120	40400	4040	8080	NA ^c	35000	NA	5100	NA	NA	189	NA	26.2	NA	
Dibenzofuran	1070	1070	428	3210	856	NA	630	NA	BD ^d	NA	NA	-41.1	NA	NA	NA	
Pyrene	7680	19200	1920	384	7680	NA	17000	NA	420	NA	NA	-11.5	NA	9.38	NA	
Diphenylamine	896	26388	448	896	224	NA	140	NA	310	NA	NA	-94.8	NA	-65.4	NA	
1,3,5-Trinitrobenzene	2000	0	4000	20000	4000	NA	43	NA	17000	NA	NA	NA	NA	NA	-15.0	NA
2,6-Dinitrotoluene	400	10000	400	200	5000	NA	8300	NA	66	NA	NA	-17.0	NA	-67.0	NA	
Benz[a]pyrene	8000	5000	5000	2000	8000	NA	7100	NA	2200	NA	NA	42.0	NA	10.0	NA	
Naphthalene	40000	40000	4000	40000	40000	NA	12000	NA	1500	NA	NA	-70.0	NA	-62.5	NA	
Phenol	50000	40000	80000	230000	80000	NR ^e	NR	NR	NR	NR	NR	NR	NR	NR	NR	
2,4-Dinitrotoluene	400	2000	10000	5000	0	NA	2000	NA	4400	NA	NA	0	NA	-12.0	NA	
1,2-Benz[a]anthracene	392	980	3920	392	784	NA	1300	NA	480	NA	NA	32.7	NA	22.4	NA	

^aSamples (acetonitrile solution) analysed GC/MS with Finnigan MAT 8430 double focusing MS.

^b $\frac{[(Assayed(A)-Spiked(O))/O]}{O} \times 100$ = Percent deviation from spiking level.

^cNA denotes not applicable or sample not assayed.

^dBelow detection.

^eNR denotes not reported.

(1) Low recoveries (less than 50%) were found for all analytes for the phase B soil sample spikes; these low recoveries were subsequently determined to have resulted from reduced sensitivity of the SFC/MS used to analyze the extracts. The reduced sensitivity resulted from the chromatographic coating being stripped from the SFC's column by the acetonitrile carrier and being carried to the MS's quadrapole area where it deposited. The SFC/MS unit was repaired for the phase C samples.

(2) The samples used for phase C were spiked using two established methods, the slurry/rotary evaporation technique and the aluminum (foil) roll technique to see if the spiking technique affected the recovery of the analytes from the soil. In the slurry technique the solvent (100 - 200 ml per 400 grams of soil) is removed at elevated temperatures (45-80°C) using a rotary evaporator. There was concern that some of the more labile analytes would be lost. In contrast, the aluminum roll technique lets the solvent (1 ml) air-evaporate. Five soil samples were spiked by the slurry technique and five were spiked by the aluminum roll techniques. The samples were spiked with the same quantities of analytes. The analyses of the spiked soil samples were performed by AWL. The samples were extracted using acetonitrile, followed by a rotary evaporation operation to reduce the quantity of extractant. For some of the analyses, the acetonitrile was replaced by dichloromethane solvent. The analyses were performed in four different ways:

- (a) Dichloromethane solution, analyzed by GC/MS with Finnigan MAT INCOS 50 MS.2
 - (b) Dichloromethane solution, analyzed by GC/MS with Finnigan MAT 8430 double-focusing MS.
 - (c) Acetonitrile solution, analyzed by GC/MS with Finnigan MAT 8430 double-focusing MS.
 - (d) Dichloromethane solution, analyzed by SFC/MS with Finnigan MAT INCOS 50 MS.
- (3) The summary of results of these tests (based on the percent of the analyte recovered from the soils) is shown in Table 3.29.

Table 3.29 Summary of Results of the phase C Soil Spiking Study.

Compound	Number	Average (percent)	Standard Deviation (percent)
1-Nitropyrene	30	114.6	32.3
Dibenzofuran	32	74.1	24.1
Pyrene	34	87.9	19.8
Diphenylamine	30	49.3	21.8
1,3,5-Trinitrobenzene	24	45.8	18.3
2,6-Dinitrotoluene	34	62.6	15.5
Benzo[a]pyrene	30	91.5	17.7
Naphthalene	34	30.8	10.7
2,4-Dinitrotoluene	28	105.5	60.8
1,2-Benz[a]anthracene	34	133.8	38.3
Average	31	79.6	25.9

Note: Eleven outlier values have been removed.

- (4) The major conclusions of the evaluation were:
 - (a) There were no significant differences between the aluminum roll and the rotary evaporation methods of preparing the spiked samples.
 - (b) There were no significant differences between the GC/MS and SFC/MS methods of analysis, using the Finnigan MAT INCOS 50 MS.
 - (c) There were no significant differences among the various GC/MS methods.
 - (d) There were no significant differences in recovery between samples.
 - (e) There were significant differences in recovery between compounds.

(f) Recoveries, on the average for each compound were less than 100 percent, the averages by compound varying from 31 percent to 134 percent, with an overall average of 80 percent.

(g) Recoveries varied considerably as indicated by the standard deviations given in Table 3.29.

(h) Phenol was not recovered from any sample. Therefore, phenol should not be used as a target analyte at least if a Soxhlet extraction procedure is used. Whether phenol would be recovered if a sonification technique is used for extraction is unknown.

(i) The results show that the analytes can be quantitatively transferred to dichloromethane from the acetonitrile used to extract the analytes from the soil. This is encouraging because it provided a means to remove the acetonitrile solvent before the sample is placed in the SFC/MS system.

(S) Detection Limits for Semivolatile Organic Analyses of Soil Samples

Zero spiked amounts were made for two compounds, 1,3,5-trinitrobenzene and 2,4-dinitrotoluene, in four different samples. The reported amounts by the various methods of analyses for these compounds (fifteen values) varied in a random way between 18 and 380 nanograms. There were no consistent differences associated with method of spiking, method of analysis, or compound. Based on these results, the detection limits for semivolatile organics for soil samples should be stated as 400 nanograms.

3.2.3.3. Battelle-Columbus Division

3.2.3.3.1 BB Test

BCD participation was limited to the BB Test. In addition to analyzing some of the test samples, they analyzed an EPA-spiked solution, the extract from two 32-L tanks initially spiked by EPA and extracted by OGC, and EPA-spiked resins (Porapak-R™ and XAD-2™) in a parallel study with AWL.

3.2.3.3.2 Field Tests

Because BCD did not participate in the major portions of the project (phases A, B, and C), no further details of its work are included in this report.

3.2.3.4. Oregon Graduate Center

3.2.3.4.1 Media

The OGC laboratory analyzed air samples contained in 6-L canisters and 32-L tanks for volatile organic compounds on the OB/OD target analyte list. EPA spiked the canisters and tanks with gases from the U.S. National Institute of Standards and Technology (NIST)-traceable cylinders.

3.2.3.4.2 BB Test

Table 3.30 presents results of the OGC analyses of canisters spiked during the BB phase. The results for the more volatile compounds were very good. The increased negative error for the less volatile compounds is attributed to the tendency of these compounds to condense in the canister when the humidity of the gas is less than 5 percent.

Table 3.30 Results of Analysis of Volatile Organic Compound Canisters at OGC*.

Spiking Compound	Spiked Level (ppb)	Reported (ppb)		Difference (percent)	
		CQ 142 ^b	CQ 338 ^b	CQ 142 ^b	CQ 338 ^b
Vinyl chloride	3.4	3.7	3.6	8.8	5.9
Bromomethane	3.6	3.8	3.6	5.6	0.0
Trichlorofluoromethane	3.8	3.7	3.6	-2.6	-5.3
Carbon tetrachloride	3.5	2.8	3.0	-20	-14
Methylene chloride	3.5	3.4	3.1	-2.9	-11
Chloroform	3.6	4.7	4.5	30	25
1,1,1-Trichloroethane	3.6	3.4	3.2	-5.6	-11
1,2-Dichloroethane	3.7	2.7	2.4	-27	-35
Benzene	3.3	2.5	2.2	-24	-33
Toluene	3.7	2.6	2.1	-30	-43
1,2-Dibromoethane	3.9	2.4	1.9	-38	-51
Tetrachloroethylene	3.8	3.3	3.0	-13	-21
Chlorobenzene	3.8	2.6	2.0	-32	-47
o-Xylene	3.7	1.8	1.0	-51	-73
Trichloroethylene	4.2	3.5	3.3	-17	-21
1,2-Dichloropropene	3.8	2.8	2.3	-26	-39
Ethylbenzene	3.6	1.9	1.2	-47	-67

*Oregon Graduate Center.

^bSample identification number.

3.2.3.4.3 Phase B

The results of spiked canisters for the phase B effort for benzene, a compound of particular interest, were very good as shown in Table 3.31.

Table 3.31 Results of OGC^a Analysis of Benzene-Spiked Canisters.

Sample	OGC Result ^b (ppb)	Spiked Concentration (ppb)	Difference (ppb)	Difference (%)
A	0.9	0.9	0.0	0
	0.8	0.9	-0.1	-11
B	1.2	1.0	0.2	20
	1.1	1.0	0.1	10
C	2.9	2.5	0.4	16
	2.6	2.5	0.1	4
D	3.4	3.0	0.4	13
	3.2	3.0	0.2	7
		Average	0.16	7.4
		Std Dev ^c	0.18	9.8

^aOregon Graduate Center.

^bEach sample was analyzed twice.

^cStandard deviation.

3.2.3.4.4 Phase C

As part of the phase C testing, EPA prepared three spiked 6-L canisters and one blank 6-L canister humidified zero air for analysis by OGC. The spiked canisters contained various levels (1.7 - 2.8 ppbv) of hydrocarbons (C₂ - C₈). The detailed results of OGC's analyses, considered excellent for the low concentrations involved, are given in Table 3.32. The results reported on the blank canister did not list any target analytes above trace levels.

Table 3.32 Results of OGC* Analyses of EPA^b Spiked 6-L Canister, phase C.

Analyte	Canister	Spiked (ppbv)			Reported (ppbv)			[(A-O)/O]x100 ^c		
		A	B	C	A	B	C	A	B	C
Ethane		1.7	2.6		1.5	3.0		-12	15	
Propane		11.8	2.8		1.9	3.1		5.6	11	
Isobutane		1.8	2.8	2.4	1.9	3.0	2.5	5.6	7.1	4.2
n-Butane		1.8	2.8	2.4	1.8	3.0	2.5	0	7.1	4.2
Isopentane		1.8	2.7	2.4	1.8	2.9	2.6	0	7.1	8.3
n-Pentane		1.8	2.8	2.4	1.8	3.1	2.6	0	11	8.3
3-Methylpentane		1.8	2.7		1.7	3.3		-5.6	22	
n-Hexane		1.8	2.7	2.4	1.7	2.9	2.6	-5.6	7.4	8.3
Benzene		1.8	2.8		1.7	2.8		-5.6	0	
n-Heptane		1.8	2.7	2.4	1.7	2.8	2.5	-5.6	3.7	4.2
Toluene		1.7	2.7		1.6	2.5		-5.9	-7.4	
n-Octane		1.7	2.7	2.4	1.7	2.7	2.5	0	0	4.2
o-Xylene		1.7	2.6		1.4	2.1		-18	-19	

*Oregon Graduate Center

^bU.S. Environmental Protection Agency

^cAssayed(A)-Spiked(O)/Spiked(O)

3.2.3.5. Lawrence-Berkeley Laboratory. No performance audit of LBL was conducted due to the extensive LBL application of XRF analytical techniques over a period of many years.

3.2.3.6. Sunset Laboratory. No performance audit of SSL was conducted because of SSL's status as the foremost U.S. laboratory conducting carbon analyses.

3.2.3.7. Lockheed Engineering and Sciences Company. No performance audit of LESC was conducted because the nature of LESC support did not lend itself to this type audit.

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SECTION 4. SUMMARY OF DATA QUALITY ACHIEVED

4.1. General

This evaluation of data quality includes an analysis of test results for estimating precision, accuracy, detection limits, and data completeness.

4.1.1. The data quality goals for the OB/OD study are summarized in Table 4.1, which was extracted from the QAPP. In most cases, these estimates have been confirmed or refined with actual data obtained from audits or results of analyses. The analyses plan for OB/OD has complicated source estimates, e.g., data completeness, by requiring an excess number of individual samples to be taken and subsequently making decisions regarding sample compositing and number of samples that would be analyzed.

Table 4.1 Precision, Accuracy, Completeness, and Detection Limit Objectives.

Chemicals/Flow Rate	Precision/Method	Accuracy/Method	Completeness	Detection Limits
Exotic organics (soil particulate)	± 0-148 ng/sample, duplicate samples (depends on analyte)*	± 50% audit samples, lab standards	90%	0.03-196 ng/ml (depends on analyte)
Exotic organics (filters)	± 0-175 ng/sample, duplicate samples (depends on analyte)*	± 50% audit samples, lab standards	90%	0.03-196 ng/ml (depends on analyte)
Metals (filters)	± 10% /duplicate samples	± 30% Al, ± 10% Si, ± 3 % all others/NIST® standards	90%	2-15 ng/cm ² (depends on analyte)
Volatile organics (6-liter canisters)	± 20% /duplicate samples	± 15-20% audit samples, lab standards	90%	0.1-0.2 ppb
Real-time gases (FWAC)	± 15% /duplicate checks	± 15% /audit samples, lab standards	90%	5.0 ppb - 1.2 ppm ^c
Flow rates	± 10% /duplicate checks	± 15% /flow standards	90%	NA ^d

*Table 2.6 contains detection limits for target analytes.

^bNational Institute of Standards and Technology.

^cCO₂ = 1.2 ppm, CO = 0.1 ppm, O₃ = 5.0 ppb, NO_x = 6.0 ppb, NO₂ = 6.0 ppb, NO = 6.0 ppb.

^dNot applicable.

4.2. Particulate Mass

4.2.1. Particulate mass was measured in all phases. In the BangBox phase, high-volume samplers were operated in the air chamber for each burn or detonation. In phases A, B, and C, high-volume-type samplers were operated when the aircraft entered the detonation or burn cloud. A number of studies have been made in estimating the precision of particle mass determinations when filters are used as a collection medium (Reference 4). SNL performed an analysis using as a basis, the error variability in the weighing of three control filters used in phase C. The standard deviation for a given weighing is estimated to be 0.3 mg. For the particulate mass on a single filter, the standard error would be 0.42 mg or 95-percent probability limits of ± 0.85 mg. When considering the combined mass on the three filters used concurrently on a given test, the 95-percent probability limits would be ± 1.5 mg. For a "dirty" explosive detonation test, the 95-percent uncertainty limits in the mass determination would be about ± 0.05 percent. However, with a relatively clean propellant burn, when a very small amount of particulate is collected, the 95-percent uncertainty limits would be from ± 10 to 15 percent.

4.2.2. Weighing accuracy was controlled by using NIST-traceable, class S weights at the beginning of each weighing cycle. These class S weights had been recently calibrated (August 6, 1990) just prior to the beginning of phase C. Because class S weights are generally accurate to ± 0.00002 gram, any departure from absolute weighing accuracy would have been inconsequential when compared to the effects of other variables in the weighing process.

4.3. Gases

Four important gaseous constituents measured in samples of the detonation and burn clouds during all tests were CO, O₃, and NO/NO₂ (all EPA criteria pollutant gases), and CO₂. SO₂, which was measured during BangBox testing, is also a criteria pollutant gas. The analyzers used for these gases had been declared as EPA-equivalent methods and had passed stringent demonstration tests with respect to detection limits, precision, and accuracy.

4.3.1. All of the performance audit checks for accuracy cited in Section 3, with one marginal exception, met the ± 10 percent requirement/goal.

4.4. Semivolatile Organics

4.4.1. General Considerations

Although both AWL and BCD performed analyses of semivolatile organics on samples taken during the BangBox test, analyses during phases A, B, and C were conducted by only AWL because of its use of the SFC-MS. Because AWL performed the preponderance of analyses throughout the entire OB/OD test sequence and precision and accuracy data for BCD have been reported previously (Reference 1), data quality presented in this report is confined to AWL.

4.4.2. Summarizing Precision Data

The following steps were taken to simplify summarizing precision data for semivolatile organics:

4.4.2.1. Precision estimates were combined for analytes analyzed by both SFC-MS and GC-MS (because the results are generally comparable).

4.4.2.2. Precision was reported as percent relative standard deviation, because precision is generally proportional to concentration level.

4.4.2.3. Precision was not listed for analytes not found.

4.4.2.4. Ranges of estimates are stated to cover all types of sample media and test materials.

4.4.3. Simplifying Precision Determinations

Precision determinations were simplified for the following reasons:

4.4.3.1. The list of target analytes was lengthy.

4.4.3.2. The lists of target analytes varied, depending on the explosive or propellant.

4.4.3.3. There were two different methods of analysis, SFC-MS and GC-MS.

4.4.3.4. Precision could vary with the sample matrix, i.e., soil, fallout, or particulate on filters.

4.4.3.5. Precision could vary with the level of concentration.

4.4.3.6. Precision cannot be estimated if the analyte is not present.

4.4.3.7. Precision can be estimated in different ways.

4.4.4. Estimates of Analytical Instrument Precision

The estimates given in Table 4.2 were obtained from duplicate injections of the analytes in measurable quantities in laboratory solvents.

Table 4.2 Instrument Precision for SFC/MS Analysis of Semivolatile Organics.

Analyte	Percent Relative Standard Deviation	
	BangBox	Phases A & B
2-Nitronaphthalene	1.5 - 7.6	1.7 - 6.9
2,4-Dinitrotoluene	1.2 - 2.0	1.4 - 7.1
2,6-Dinitrotoluene	1.0 - 11.7	1.6 - 4.9
4-Nitrophenol	5.4 - 15.8	
Dibenzofuran	0.9 - 8.5	
N-Nitrosodiphenylamine	1.5 - 10.6	
Benz[a]anthracene		
Benzo[a]pyrene		
1-Nitropyrene	1.1	
2,4,6-Trinitrotoluene	2.9 - 7.7	1.4 - 3.7
Naphthalene	1.0 - 5.2	1.5 - 6.7
1-Methylnaphthalene	7.5	
2-Methylnaphthalene	7.5	
1,3,5-Trinitrobenzene	0.9 - 5.6	1.3 - 4.7
Biphenyl	0.7 - 5.0	
Phenanthrene	1.2 - 5.9	1.6
Pyrene	2.2 - 3.1	2.0 - 3.6
2,5-Diphenylazazole	5.3 - 8.2	
1,1,3-Trimethyl-3-phenylindane	9.5 - 12.9	
Nitroglycerin		4.1
Phenol		2.2
2-Nitrodiphenylamine		2.4

4.4.5. Interpretation

As noted from the table above, the agreement between analyses of duplicate extracts is very good, the maximum value reported being 15.8 percent. Most of the differences may be the result of small sample variability. The average value for the percent relative standard deviation is 4.5 percent. Since each estimate has been made from only two values, i.e., duplicates of the same extraction, the 95-percent probability limits for the relative standard deviation are 0.1 percent and 10.1 percent, limits that include practically all of the above data. It is reasonable to conclude that the precision for all of the analytes is about 5-percent relative standard deviation.

4.5. Semivolatile Organics in Soil and Filter Samples

4.5.1. Alpine West Laboratories performed a special study in November 1990 in which the originally reported analyses of background soil, ejecta, and fallout from Phase B were compared with (1) the results from extracting and analyzing, in duplicate, unextracted portions of those samples that had been stored under refrigeration and (2) reanalyses of the original extracts which had been stored under refrigeration. No significant differences were observed, except for the background soil, in which case the original reported results for several target analytes were somewhat higher than the results from the duplicate samples and retained extracts. Table 4.3 presents the results for those target analytes and samples for which all three data sets had reported data. In the other cases, one or more of the reported results was "not detected".

4.5.2. Results

Using the results to estimate the overall precision of the method, the coefficient of variation averaged about 60 percent. Such results are considered good for analyses of soil at the ng/g (ppb) level.

4.5.3. Estimates of Precision during phase C

Estimates of precision for the analysis of semivolatile organics by SFC/MS and GC/MS were obtained in several ways:

Table 4.3 Verification of Phase B Soil Analyses.

Analyte (all values ng/g)	Sample* A	Original Analysis	Recent Duplicate Soil Analysis	Recent Analysis of Extract	Standard Deviation	Average (all 3 values)	CV, percent
2,4-Dinitrotoluene	A	5.6	2.5	6.8	2.22	4.97	45
	C	32.0	76.0	4.2	36.20	37.40	97
	E	2.2	6.7	9.0	3.46	5.97	58
2,4,6-Trinitrotoluene	A	2000.0	750.0	560.0	782.00	1103.00	71
	B	237.0	180.0	260.0	41.20	226.00	18
	C	45.0	34.0	150.0	64.00	76.30	84
1,3,5-Trinitrobenzene	E	12.0	14.0	29.0	9.29	18.30	51
	A	140.0	65.0	44.0	50.50	83.00	61
	Naphthalene	A	47.0	17.0	8.2	20.30	24.10
	B	46.0	38.0	14.0	16.70	32.70	51
	C	74.0	120.0	69.0	28.10	87.70	32
	D	2.4	2.2	7.5	3.00	4.03	75
Benz[a]anthracene	E	17.0	17.0	29.0	6.93	21.00	33
	B	6.8	7.2	3.4	2.09	5.80	36
	C	6.4	2.8	13.0	5.17	7.40	70
Pyrene	B	9.2	5.5	1.3	3.95	5.33	74
	C	36.0	9.6	7.1	16.01	17.60	91

*Sample Identification

A is a Background Sample.

B and D are Ejecta Samples.

C and E are Fallout Samples.

4.5.3.1. Duplicate Analyses of Spiked Soil Samples Spiked at the Same Level

4.5.3.1.1 Because the EPA-spiked soil studies for phases A and B involved microgram quantities (i.e. considerably more than were being encountered with test samples collected in the field), an additional EPA-spiked soil study was conducted for phase C, but at nanogram quantities (i.e. at levels being encountered with test samples). Although the spiked soil study was being conducted primarily for purposes of estimating accuracy, or percent recovery, (See Section 3.2.3.2.2 for the results), some information could be gained from the results with respect to precision since the same spike levels for all compounds were replicated in different samples.

4.5.3.1.2 The compounds included in the study are listed in Table 4.4. Most levels used in the spiking study, including zero amounts, were replicated at least once. There were 22 duplicates, 5 triplicates, 7 quadruples, and 1 sextuplicate. Further, all of the samples were analyzed by three different methods, and some by four different methods.

4.5.3.1.3 Among the replicates, the repeatability on a percent recovery basis was not significantly affected by the spiking level. The average standard deviation of repeatability in terms of percent recovery was 23 percent, varying between 2 and 41 percent for individual compounds and levels. There were no significant differences in precision between compounds. From this overall precision estimate, 95% confidence limits for individual reported results would be ± 46 percent. This computation makes no consideration for the average recovery level being significantly less than 100 percent. However, it should be recognized that this estimate includes some possible sample-to-sample variability in the spiking process. It does include any variations introduced by the extraction process since extraction is a part of the total analysis method.

4.5.3.2. Regression Analysis of Spiked Soil Samples Analyses,

4.5.3.2.1 Regression analyses were performed for each compound except for phenol, which was not recovered from any of the samples. The scatter of the individual data points about the regression line, when the analyzed, or reported, values are plotted against the spiking level, is a measure of precision of the analysis. The "standard error of the estimate" varied between 111 ng for dibenzofuran to 2567 ng for naphthalene. The pooled value across all ten compounds was 1491 ng.

Table 4.4 Precision (Relative Standard Deviation) of Analyses by GC/MS and SFC/MS for Semivolatile Organics for Phase C.

Semivolatile Organic	Duplicate Spiked Soil	Duplicate Analyses Soil and Filters
2-Nitronaphthalene		21
2,4-Dinitrotoluene	56	8
2,6-Dinitrotoluene	16	45
Dibenzofuran	13	59
N-Nitrosodiphenylamine		97
Benz[a]anthracene	35	83
Benzo[a]pyrene	14	113
1-Nitropyrene	27	25
2,4,6-Trinitrotoluene		14
Naphthalene	10	43
1,3,5-Trinitrobenzene	14	61
Pyrene	19	26
2-Nitrodiphenylamine		54
Diphenylamine	15	84

This value is an average over all compounds and levels and does not reflect the fact that for most chemical analyses, the precision variability in terms of the reported units increases with an increase in level. Consequently, the following data plot was made of the standard error of estimate versus an 'average' (i.e. weighted) spike amount.

4.5.3.2.2 This data plots showed that the standard error of estimate increases linearly up to a level of about 12,000 ng, where the standard error is 2600 ng, or 22 percent. The precision estimate then in terms of a coefficient of variation (relative standard deviation) for all levels from about 400 ng up to 12,000 ng is about 22 percent. Corresponding 95 percent confidence limits for individual reported values would be ± 44 percent. This value agrees very closely with the ± 46 percent given above in Section 4.5.3.1.3.

4.5.3.3. Duplicate analyses of extracts of test soil and filter samples collected during phase C.

4.5.3.3.1 During the analyses of soil and filter samples, a number of duplicate analyses were made

by GC/MS and SFC/MS. A sample of these results were evaluated. The following numbers of samples were considered:

Filters, by GC/MS:

RDX Samples - 3

Yellow D Samples - 7

Soil, By GC/MS:

RDX Samples - 3

Yellow D Samples - 2

Soils, by SFC/MS:

RDX Samples - 4

Yellow D Samples - 4

4.5.3.3.2 The reported results for these duplicate analyses included all of the ten spiked and reported compounds plus four additional compounds. The results confirmed the increasing precision variability with increased level of the analytes. Most all of the results were below 400 ng, with some results between 400 and 1800 ng. The slope of the relationship between absolute percent difference between duplicates and the average analyte content was 0.49. The standard deviation of signed percent differences for results below 400 ng was 97 percent. This value would indicate 95 percent confidence limits of individual reported values of ± 136 percent. This determination includes numerous pairs of data where one of the values was reported as zero, and other pairs averaging less than 400 ng. Above 400 ng, the average percent absolute difference between duplicates is about 35 percent. The standard deviation of signed percent differences (or relative standard deviation) is 39 percent. This value would indicate a 95 percent confidence limit of ± 55 percent for individual reported values. The standard deviation of signed differences is 413 ng.

4.5.3.4. Interpretation

A summary of the results from the above sections are given in Table 4.4. Thus, the ± 46 percent limits from paragraph 4.5.3.1.3, the ± 44 percent limit from paragraph 4.5.3.2.2, and the ± 55 percent limits of paragraph 4.5.3.3.2 are all in very consistent agreement with the data of Table 4.3 which reports the results of soil samples of phase B. Note that these limits do not apply to amounts

less than 400 ng because the relative standard deviation increases drastically as the amounts become smaller.

4.6. Elements

4.6.1. Measures of Precision

Lawrence-Berkeley Laboratory analyzed particulate filters by XRF for various elements in the BangBox test and in Phase C. They reported the measures of precision for 19 elements on their control filters (Table 4.5).

4.6.1.1. The values were compiled from over 40 separate analyses for element concentrations on

Table 4.5 Precision of Elemental Analyses by XRF*.

Element	Coefficient of Variation (%)
Sulfur	0.7
Potassium	1.0
Calcium	1.0
Titanium	2.9
Manganese	2.7
Iron	1.0
Nickel	2.0
Copper	7.5
Zinc	0.7
Rubidium	1.8
Strontium	1.1
Lead	0.9
Zirconium	5.6
Molybdenum	2.0
Silver	4.3
Cadmium	1.9
Tin	2.4
Antimony	2.6
Barium	5.5

filters varying between 1 and 10 $\mu\text{g}/\text{cm}^2$. The precision values given above are considered to be good state-of-the-art values with Teflon™ filters. However, in phase C, when Teflon™ impregnated glass fiber (TIGF) filters were used, the impurities in the glass (Zn, Rb, Sr, Ba) interfered considerably with the precision of the analysis. The reported precision for the analysis of Cu and Pb were about 10 percent.

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SECTION 5. SUMMARY AND CONCLUSIONS

5.1. General

The QA program used during OB/OD testing and analyses was adequately planned and executed, resulting in data of sufficient quality to determine which methodologies, instruments, and equipment should continue into item- and site-specific testing.

5.2. Letters of Instruction

The LOI's developed during OB/OD testing and analyses proved to be an excellent vehicle for standardizing and documenting evolving procedures, techniques, and applications. Their preparation and availability before initiation of activities facilitated integration of a multitude of diverse, but interrelated, technical operations. The LOI's were treated as living documents and updated by each activity when procedures were modified, so that the final procedures used can be identified and followed as the study advances into its next testing phase. The few exceptions to LOI development, e.g., analysis by XRF, involved procedures that had been developed, tested, and accepted by the scientific community many years prior to planning for this study and which required no further refinement.

5.3. Sample Tracking and Sample Security

The system established by ELI permitted positive sample identification, handling, and location throughout the project. The system allowed for integration of separate systems in-place at analytical laboratories which met OB/OD accountability objectives. In several instances, auditors noted that samples were not constantly maintained in a locked container, but concluded that sample integrity did not appear to have been jeopardized.

5.4. Systems Audits

The systems audits as conducted by EPA and ELI were complementary and permitted the PM to maintain constant control over application of procedures and technologies during all testing and

analytical activities. Auditors held professional credentials appropriate to their mission and conducted their visits in a thorough and expeditious manner. The overwhelming majority of these audits confirmed that good practices were being followed and concluded that the desired test/analytical results were attainable. These audits also identified potential problem areas and facilitated remedial action before sample or data quality was threatened. In the instance where an audited organization did not pursue action recommended by the auditors following each of several audits, equipment failure resulted, with consequential delays during field testing (data quality was unaffected).

5.5. Performance Audits

5.5.1. Instruments and Equipment

The EPA conducted all performance audits throughout the OB/OD study. Auditors determined that instruments and equipment, when audited, met or exceeded accepted standards for accuracy and response.

5.5.2. Spiked Samples

5.5.2.1. The EPA spiked soil samples taken from the field testing site, canisters provided by the laboratory conducting analyses for volatile organic compounds, and resin samples provided by the laboratories conducting analyses for semivolatile organic compounds. The EPA also provided spiking solutions to the latter laboratory for internal soil spiking. Following analyses and assays by the audited organizations, EPA compared laboratory results to known values. The correlation of findings to known values was high and indicated that analytical results of actual test samples would be of sufficient accuracy to meet study objectives.

5.5.2.2. The spiking of soil for the BangBox phase and phases A and B were made at μg levels. Because many of the analyses of actual test samples were in ng levels, for phase C an additional spiking study was conducted. The analyses showed average percent recoveries varying from 29 percent to 90 percent for the compounds spiked. Phenol was not recovered in any amount for spiked amounts up to 120,000 ng. It is speculated that the phenol is lost completely in the spiking

preparations and/or in the rotary evaporation operation following extraction of the samples for analysis. Consequently, considerations should be given to corresponding adjustments of the OB/OD results.

5.5.3. Reports

Environmental Labs Inc., reviewed the BangBox Thermal Treatment Emission Study Final Report and found that it properly reflected test conduct and results of analyses. The report for field testing has not yet been reviewed by any QA agency.

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SECTION 6. RECOMMENDATIONS FOR PHASE D TESTING

The following recommendations are based on experience gained from the BangBox test and subsequent field test phases A, B, and C. They primarily address QA activities, but have some bearing on general planning for future OB/OD testing.

6.1. Laboratory Selection

6.1.1. Laboratories under consideration should fully demonstrate the capability of performing routine analyses of the type and method desired.

6.1.2. Personnel, equipment, procedures, and quality control practices should be integrated into the selection process. Systems and performance audits should be conducted prior to contract award, unless a sole source contract, based on previous experience with the laboratory, is desired.

6.1.3. All contracts to supporting laboratories should stipulate that calibration and internal QC data will be included with sample analysis data and that calibration and QC data should be presented in time-sequence relationship to sample analyses.

6.1.4. All laboratories should be required to perform duplicate analyses of some samples on the same day and some duplicate analyses with a one-day minimum separation between analyses.

6.2. Documentation

6.2.1. All TDP's, QAPP's, and LOI's should be fully reviewed and approved before actual sampling, testing, or analyses begin.

6.2.2. Procedural and operational changes should be prohibited, unless approved by the PM. Side-by-side studies should be performed to compare the existing procedure with the proposed procedure.

6.2.3. When procedural or operational changes are approved, related documentation should be revised and reissued immediately.

6.2.4. All documentation should be dated and should clearly identify the author by name and organization.

6.3. Operations

6.3.1. Attention to detail must be stressed to all persons engaged on project activities.

6.3.2. The TSC concept should be continued, and the study QA director should remain a member of the TSC.

6.3.3. The TDP's, QAPP's, and the LOI's to provide for adequate replication of tests (materials and conditions), sampling, and analysis (chemical, weighing, and flow measurement) within the day and between days.

6.3.4. If not accomplished prior to contract award to laboratories, both systems audits and performance audits should be accomplished as soon as possible after award. Systems audits and performance audits should also be conducted during the time actual OB/OD samples are being processed.

6.4. Quality Assurance Assignments and Responsibilities

6.4.1. The QA responsibility within each organization should be assigned to the individual with the best qualifications. This individual should prepare the internal QC plan and periodically prepare reports on organizational QA activity including quantitative data quality information.

6.4.2. The sampling organizations, TSC and QA director should jointly develop the sample assignment/numbering system. The complete system, including sample forms and sample tracking forms should be included in the QAPP and in the LOI's of the sampling organizations.

6.4.3. The sampling organizations should be responsible for delivering their samples to the laboratory. Under normal circumstances, QA personnel should not transport or deliver samples.

6.4.4. Both the sampler/sender and the recipient should notify the QA sample-tracking agent when samples are transferred.

6.5. Quality Assurance Project Plan

6.5.1. The QA director should be responsible for preparing the QAPP.

6.5.2. The plan should be approved by the PM prior to testing.

6.5.3. The plan should include (1) the sample numbering system in as complete a mnemonic manner as possible, (2) the sample control system, (3) complete instructions for preparing and revising LOI's, (4) complete instructions for document control, and (5) archival instructions for each type of record involved.

6.6. Nomenclature

6.6.1. A single definition and procedure for determining detection limits and limits of quantification should be adopted and included in both the QAPP and respective LOI's.

6.6.2. All numerical analytical results should be reported, even if below the limits of detection or limits of quantification.

6.6.3. Blanks (instrument, filter, soil, field, solvent, solution, reagent, background, or travel) and their use in correcting or adjusting raw data should be precisely identified.

6.7. Letters of Instruction

6.7.1. Operational organizations should complete their LOI's in time for the PM and QA director to complete their review before testing begins.

- 6.7.2. Originators should precisely follow the format specified by the QA director.
- 6.7.3. LOI numbers should not be reassigned to a different topic or procedure.
- 6.7.4. Originators should carry out preventive maintenance actions at the frequencies recommended by instrument and equipment manufacturers.
- 6.7.5. Project personnel should develop a spare-parts inventory for all critical equipment and specify stocking levels in their LOI's.
- 6.7.6. The EPA should consider developing LOI's for spiking soil, canisters, and filters.

6.8. Laboratory Reporting and Control

- 6.8.1. Each laboratory should maintain statistical quality control charts for recording routine OB/OD-related quality control data.
- 6.8.2. All laboratories should keep laboratory notebooks in general accordance with the recommendations of "Writing the Laboratory Notebook", by H.M. Kanare (Reference 5).
- 6.8.3. If, possible, laboratory analytical results should be carried out and reported to three significant digits, even if the third digit is considered an estimate or interpolated value.

Reason: Some loss in the estimation of precision is incurred when data are always rounded to only two significant digits. Only two significant digits have been used as a rule for GC/MS and SFC/MS data. For example, duplicate results could be reported as 30,000 and 30,000 nanograms, and in another case the two results could be 25 and 25 nanograms. In the first case, a one-digit change would be only 0.0033 percent, whereas in the second case it would be a 4.0 percent change.

6.9. Notebooks

- 6.9.1. All key personnel should keep notes concerning OB/OD in bound laboratory-type

notebooks.

6.9.2. Each organization, field or laboratory support, should maintain a project notebook for the OB/OD project, and additional logbooks for each major instrument.

6.9.3. When possible, project personnel should use preprinted forms permanently secured in notebooks or logbooks.

6.10. Systems Audits

6.10.1. Auditors should rely upon preplanned audit checksheets (which provide for narrative entries) and/or questionnaires.

6.10.2. Independent specialists should be engaged to augment the audit team if the auditor is not fully familiar with the technical area involved.

6.10.3. The auditor should conduct an exit interview at the completion of each systems audit and summarize all major findings. The QA agency should provide a formal written report to the audited organization within 10 working days after the audit.

6.10.4. Meteorological and photographic support should be included in future systems audits.

6.10.5. Auditors should gather documents and photographs to support the audit report.

6.11. Performance Audits

6.11.1. Prior to the beginning of any future OB/OD studies involving the analysis of soils or filters, a thorough study should be performed to investigate the various factors involved in spiking and analyzing soils (and filters) to determine the specific locations, causes, and extent of losses of semivolatile organics.

6.11.2. Prior to the beginning of any future OB/OD studies, special studies should be conducted

by the chemical laboratories involved to determine detection limits according to an expert committee agreed-upon plan.

6.12. Corrective Action

6.12.1. The auditor should be limited to identifying actual or potential problem areas, but should be given discretionary authority to suggest corrective actions and provide informal technical assistance.

6.12.2. The audited organizations should be held responsible for isolating the basic causes or problems and taking appropriate corrective actions.

APPENDIX E. CONSOLIDATED ABBREVIATIONS

ACGIH	American Conference of Governmental Industrial Hygienists
AEHA	U.S. Army Environmental Hygiene Agency, Aberdeen Proving Ground, Maryland
AFB	Air Force Base
AMC	U.S. Army Materiel Command, Alexandria, Virginia
AMCCOM	U.S. Army Armament, Munitions and Chemical Command, Rock Island, Illinois
amino-PAH	aminopolycyclic aromatic hydrocarbons
ANOVA	analysis of variance
AP	ammonium perchlorate
APS	aerodynamic particle sizer
ASASP	active scattering aerosol spectrometer probe
AWL	Alpine West Laboratories, Provo, Utah
BB	BangBox
BCD	Battelle Columbus Division, Columbus, Ohio
BD	target analyte not found in concentrations above detection limits
BYU	Brigham Young University, Provo, Utah
CAA	Clean Air Act
CDD	chlorinated dibenzodioxin
CDF	chlorinated dibenzofuran
CI-SIM	chemical ionization, selective-ion monitoring
CSI	Columbia Scientific Instruments
C·V	concentration times cloud volume method
CWA	Clean Water Act
DMC	Data Management Center
DMPS	differential mobility particle sizer
DoD	Department of Defense
DPG	U.S. Army Dugway Proving Ground, Dugway, Utah
EC	electron capture <i>or</i> elemental carbon
ECD	electron capture detector
EDAX	energy-dispersive X-ray analysis
EER	Energy and Environmental Research Corporation, Irvine, California

EF	emission factor(s)
EI	electron impact
EI-MS	mass spectrometer used in the electron impact ionization mode
EI/MS	electron impact ionization/ mass spectrometry
EIS	environmental impact statement
ELI	Environmental Labs, Incorporated, Provo, Utah
EOD	explosive ordnance disposal
EPA	U.S. Environmental Protection Agency
EPO	Environmental Protection Office, U.S. Army Dugway Proving Ground, Dugway, Utah
ER	expansion ratio
FID	flame ionization detector
FSSP	forward scattering spectrometer probe
FTIR	Fourier Transform Infrared Spectrometry
FWAC	fixed-wing aircraft
GC	gas chromatograph(y)
GC-ECD	gas chromatography with an electron capture detector
GC-FID	gas chromatography with a flame ionization detector
GC/MS	gas chromatography/mass spectrometry
GLP	good laboratory practices
HE	high explosive
HMX	octamethylenehexanitramine
HNBB	hexanitrobibenzyl
HRGC/HRMS	combined capillary column gas chromatography/high resolution mass spectrometry
HS	high-speed
LASD	Los Angeles Sheriff Department
LBL	Lawrence Berkeley Laboratory, Berkeley, California
LC	liquid chromatography
LOD	limit of detection
LOI	letter(s) of instruction
MR	multiple range

MRI	Midwest Research Institute, Kansas City, Kansas
MS	mass spectrometry (or mass spectrometer)
MSA	Mine Safety and Appliance Company
NA	not targeted for analysis <i>or</i> not applicable
NASA	National Aeronautical and Space Administration
NATICH	National Air Toxics Information Clearinghouse
NBS-SRM	National Bureau of Standards (now NIST)- Standard Reference Material
ND	no data <i>or</i> detection limit not determined
NEPA	National Environmental Policy Act
NF	not found in the sample matrix <i>or</i> not determined
NIST	National Institute of Science and Technology
nitro-PAH	nitropolycyclic aromatic hydrocarbons
NIOSH	National Institute for Occupational Safety and Health
NOSIH	Naval Ordnance Station, Indian Head, Maryland
NO _x	nitrogen oxides
NS	not sampled
OB	open burning
OB/OD	open burning/open detonation
OC	organic carbon
OD	open detonation
OGC	Oregon Graduate Center, Beaverton, Oregon
OSHA	Occupational Safety and Health Administration
PAH	polycyclic aromatic hydrocarbons
PANH	polycyclic aromatic nitrogen heterocycles
PAOH	polycyclic aromatic oxygen heterocycles
PCDD	polychlorinated dibenzodioxins
PCDF	polychlorinated dibenzofurans
PETN	pentaerythritol tetranitrate
PEP	propellants, explosives, and pyrotechnics
PIC	products of incomplete combustion
PICI/SIM	Positive ion chemical ionization/selective ion monitoring
PID	photoionization detector

PIP	product improvement program
PM	program manager
PMS	Particle Measuring Systems, Inc.
PUF	polyurethane foam
QA	quality assurance
QA/QC	quality assurance/quality control
QC	quality control
QAA	quality assurance agency
QAPP	quality assurance project plan
QAU	quality assurance unit
RCRA	Resource Conservation and Recovery Act
RDX	hexamethylenetrinitramine
REMB	Research Monitoring and Evaluation Branch of USEPA
RFD	Reno (Nevada) Fire Department
RIC	relative ion count
RSD	relative standard deviation
RTP	Research Triangle Park, North Carolina
SDPDA	Special Defense Property Disposal Account
SEM	scanning electron microscope/microscopy
SFC	supercritical fluid chromatography
SFC/MS	supercritical fluid chromatography/mass spectrometry
SF ₆	sulfur hexafluoride
SIM	selected-ion monitoring (or selective-ion monitoring)
SNL	Sandia National Laboratories, Albuquerque, New Mexico
SOP	standing operating procedures
SS	stainless steel
SSC	stainless steel canister
SSL	Sunset Laboratory, Forest Grove, Oregon
STEL	short-term exposure limit
STP	standard temperature and pressure (25°C and 760 torr)
TCD	thermal conductivity detector
TDP	test design plan

TEAD	U.S. Army Tooele Army Depot, Tooele, Utah
TECO	Thermo Electron Instruments (Company)
TECOM	U.S. Army Test and Evaluation Command, Aberdeen Proving Ground, Maryland
THC	total hydrocarbon
TLV	threshold limit values
TNT	2,4,6-trinitrotoluene
TSC	technical steering committee
TSP	total suspended particulate
TWA	time-weighted average
USATHAMA	U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, Maryland
UV	ultraviolet
VOC	volatile organic compounds
VOST	semivolatile organic sampling train
VSDM	Volume Source Diffusion Model
XRF	X-ray fluorescence <i>or</i> X-ray fluorescence spectrometer

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